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on essential oils aromatic chemicals and related materials

Courtesy of Toichi Kuraoka

THE LARGEST CAMPHOR TREE IN JAPAN

ANNUAL REPORT

ON ESSENTIAL OILS
AROMATIC CHEMICALS
AND RELATED MATERIALS

1947-1948

SCHIMMEL & CO., INC.
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ESSENTIAL OILS

ANISE IN BLOOM.

Essential Oils and Related Materials

Commercial and Scientific Developments

The literature published during 1947 and 1948 was somewhat richer in contributions relating to the perfume industry than the years immediately preceding. Many research projects of varying nature were brought to a conclusion and the results in some cases were of considerable practical importance to the industry while in others they added new facts of theoretical interest to the ever-growing fund of information on essential oil chemistry.

We mention here a few of the more significant developments among those reported in this section. Jasmone, a ketone which is familiar as a constituent of the oil of jasmine, was found to occur also in peppermint oil *from* Mentha piperita *L. The parent substance of* garlic oil *was isolated and identified as a hitherto unknown amino acid and the aromatic volatile oil obtained from the roots of* couch grass *was identified as a benzenoid hydrocarbon having a highly unsaturated side chain. Analysis of* cabreuva oil *and the wood oil of* Myroxylon Pereirae *revealed that these Brazilian oils are rich in nerolidol, a compound useful in perfumery. The analytical studies on* lavandin oil, *Florida* orange oil *and Oregon* peppermint oil *represented useful contributions to the analytical literature in view of the present commercial importance of these oils.*

In developments relating to new sources of essential oils, the shrub Clausena anisata, *which yields an oil rich in anethol, was grown experimentally in Java.* Cymbopogon densiflorus, *the blossoms of which give good yields of a perfume oil, was cultivated successfully in the Belgian Congo. In Texas the cultivation of* goldenrod *as a source of an essential oil of anise-like character was shown to be economically feasible.*

Aeolanthus Graveolens Oil. The flowers and leaves of *Aeolanthus graveolens* (*Labiatae*), a plant which is widely distributed in Northern Rhodesia and Tanganyika, have a powerful rose-like odor. Naves[1] examined a sample of the essential oil distilled from the entire plant in the flowering stage and reported the constants as follows: d^{20}_4 0.8859, $n^{20}D$ 1.4750, αD — 0.34°, acid value 1.4 and ester value 11.2. The oil contained 72.5% of alcohols and 9.9% of carbonyl compounds as $C_{10}H_{16}O$. Geraniol is the principal constituent of *A. graveolens* oil while nerol is present in a much smaller amount. (Cf. Bull. Imp. Inst. 33: 449, 1935.)

Agathosma Gnidioides Oil. Smith and Roux[2] investigated the essential oil of *Agathosma gnidioides* Schlechter, a shrub belonging to the *Rutaceae* which is found in South Africa along the southeastern portion of the Cape. The natives of the region use an infusion of the leaves as a stomachic and febrifuge. The oil, after evaporation of the more volatile constituents, has a pungent and nauseating odor due to the presence of a sulfur compound. The fresh leaves in winter yielded 1.0% of oil having d^{15}_{15} 0.837, $n^{20}D$ 1.4759 and $|\alpha|^{20}D$ — 0.87°. The yield in summer was only 0.6%. The sulfur compound, $C_{10}H_{18}S_4$, was isolated by adding sodium to the high-boiling fractions of the oil and treating the reaction product with iodine, thus

breaking and regenerating a sulfur-to-sulfur linkage. The compound was obtained as an optically inactive oil of high boiling point and d^{15}_{15} 1.096. On the basis of the oxidation products it appeared to be *bis*-(1-pentenyl-2) tetrasulfide, $(CH_3CH_2CH{:}CHCH_2)_2S_4$, a compound not previously reported in the literature. The composition of *A. gnidioides* oil was found to be approximately as follows: myrcene 60%, *l*-β-pinene 5%, *l*-limonene and dipentene together 4%, *d*-linalyl isobutyrate 25%, *bis*-(1-pentenyl-2) tetrasulfide 5% and a free butyric acid (probably isobutyric), less than 0.1%. The oil also appeared to contain insignificant amounts of *d*-linalool, methyl salicylate and salicylic acid. (For the composition of sea buchu oil from another species of *Agathosma*, see Schimmel Report 1916, 56.)

Angelica Leaf Oil. Ramstad[3] reported that angelica leaves which had been gathered from plantings in the vicinity of Oslo, Norway, yielded 0.007% of an essential oil having the following properties: *d* 0.868, $\alpha^{18}D$ −46°, $n^{22}D$ 1.477, saponification value 11, and saponification value after acetylation 138. The odor of the oil was less pleasing than that of angelica seed oil.

Anise Oil. ***See also*** **Clausena Anisata Oil.** The United States imported 221 tons of anise oil valued at $321,700 from China in 1947 and 111 tons valued at $142,600 in 1948.

At the beginning of 1947 the wholesale price of anise oil in New York was approximately $1.00 per lb., but by the third quarter of the year it had declined to $0.60. During 1948 the price of anise oil remained fairly steady at $0.75–0.85 per lb.

A chemical method for the determination of anethol in anise oil, based on the addition of iodine monochloride to the ethylenic linkage, was described by Mori[4]. The method is sufficiently accurate for the commercial evaluation of anise oil although any minor constituents of the oil which have ethylenic bonds in the molecule are determined along with the anethol.

To carry out the determination, a weighed sample of anise oil is dissolved in benzene of analytical grade and an aliquot of the solution is treated with an excess of a standardized solution of iodine monochloride. The anethol is quantitatively converted to α-iodo-β-chloropropyl-*p*-methoxybenzene. To determine the amount of iodine monochloride remaining, the solution is treated with potassium iodide which reacts with the excess reagent to form an equivalent amount of free iodine. Standard thiosulfate solution is added in excess and titrated back to the end point with standard iodine solution using starch as the indicator. From the results of the titration the amount of iodine monochloride which reacted with the anise oil and the percentage of anethol in the oil can be calculated.

Araucaria Excelsa Oil. Briggs and Taylor[5] isolated isophyllocladene from the essential oil of *Araucaria excelsa* or Norfolk Island pine. The isomeric diterpene, phyllocladene, had previously been identified as a constituent of the oil (J. Soc. Chem. Ind. 60: 222, 1941).

Artemisia Oils. Gol'mov *et al.*[6] distilled an essential oil of camphoraceous odor from the fresh plants of *Artemisia austriaca*. The properties of the oil were

as follows: d^{20}_4 0.9132, $n^{20}D$ 1.4631, $[\alpha]D$ + 0.78°, acid value 3.2, ester value 17.5 and ester value after acetylation 70.2. Examination of different fractions of the oil revealed the presence of cineol, phellandrene, thujone and thujyl alcohol. The residue from fractional distillation was found to contain high fatty acid esters of thujyl alcohol including thujyl stearate and palmitate.

Warriner[7] examined a sample of the oil distilled from *Artemisia Californica*, a plant which grows wild on California range-land. The physical constants of the oil were as follows: d^{25} 0.9348, $n^{25}D$ 1.4688 and $\alpha^{20}D$ 17°13′. The oil contained 24.9% esters as thujyl acetate, 12.8% thujyl alcohol and 0.17% free acids.

Parihar and Dutt[8] examined the essential oil of *Artemisia scoparia* Waldst. and Kit. a medicinal plant of India. The dried flowering herb was extracted with alcohol and the extract, after separation of a lactone which crystallized from it, was subjected to steam distillation. A green oil of refreshing odor was obtained in a yield of 0.75% based on the dried herb. The oil was optically inactive and had n^{23} 1.5220, d^{23} 0.9497, saponification value 77.28 and acid value 6.3. The main constituent of the oil was a new sesquiterpene $C_{15}H_{24}$ which showed b.p. 252–254°, d^{15} 0.9166, n^{18} 1.5225 and gave a nitrosite m.p. 111° and nitrosate m.p. 216°. This hydrocarbon, which was named "scoparilene," made up approximately 80% of the oil. Eugenol was present in the oil to the extent of 9%.

The lactone which crystallized from the alcohol extract melted at 146° and had a pleasing odor. The substance had the molecular formula $C_{16}H_{20}O_5$, the molecule being characterized by α-unsaturation and the presence of a ketone group and two methoxyl groups. The name "scoparin" was assigned to the lactone.

Asplenium Lamprophyllum. The presence of methyl salicylate in the fern *Asplenium lamprophyllum* was demonstrated by Briggs and Taylor[9]. This ester has been found in only one other species in the whole order of ferns. Apparently methyl salicylate occurs in *A. lamprophyllum* in the free state and not in the form of a glycoside since the odor is apparent immediately when the stem is broken.

Basil Oil. Analyses of sweet basil oil distilled in California from *Ocimum basilicum* of undetermined variety were reported by Finney and Warriner[10]. In the 1947 season, 6.5 acres of sweet basil yielded 65 lb. of oil in July and 105 lb. from a second cutting in October. Table I shows the properties of these two lots of oil and of the oil distilled in July 1946.

Table I.

Properties of Sweet Basil Oil from California

	Date of Distillation		
	July 1946	July 1947	October 1947
d^{25}	0.906	0.909	0.915
n^{25}	1.4830	1.4835	1.4822
$\alpha^{25}D$	−10°30′	−13°	−10°
Acid value	4.3	4.6	2.4
Ester value	26.4	25.4	4.9
Ester value after acetylation	252.4	192.0	222.0
Eugenol	15.0%	15.8%	14.3%
Methyl chavicol[a]	6.53%	8.67%	25.43%
Linalool	61.2%	45.82%	59.71%
Solubility in 80% alcohol	1:1	1:1.25	1:1

[a] From total methoxyl minus eugenol methoxyl.

The odor of the California sweet basil oil, while pleasing, has less of the anise character than sweet basil oil of European origin because of the lower content of methyl chavicol. There is a sharp note in the odor which can be eliminated by distilling off the first 10% of the oil.

Bay Oil. Distillers of bay oil can store the leaves for a month before distilling them without significant losses of oil or phenols, according to work done by Arrillaga and Jones[11] at the Puerto Rico Experiment Station. It was found that the yield and the phenol content of the oil reached a maximum after eight days of storage, declined during the next week and then remained fairly constant. No mold developed on leaves stored for four weeks in open or closed bags, but those kept in open bags gave somewhat higher yields.

Bergamot Oil. The 1947-48 bergamot harvest in southern Italy was generally good although some fruit was damaged by damp weather or insects. Production of bergamot oil amounted to 165 metric tons as compared with 138 tons for the 1946-47 season. In the spring of 1948 there was a severe drought in the citrus region and the trees were late in flowering and had relatively few blossoms. Consequently the bergamot crop which was harvested beginning in November was smaller by about one-fourth than the crop of the preceding season.

Exports of bergamot oil from Italy for the years 1936 through 1948 are shown in Table II. The quantity of the oil exported in 1947 (102 tons) was less than half that exported in 1946 when the demand was unusually great following the end of the war. Exports of bergamot oil in 1948 amounted to 150 tons and the value was estimated at 646,000,000 lire. The United States imported 17 metric tons of Italian bergamot oil in 1947 and 77 tons in 1948.

The wholesale price of bergamot oil on the New York market fluctuated between $5.00 and $4.40 per lb. in 1947. During the first half of 1948 there was little demand for the oil and the price dropped to $3.70 per lb.

TABLE II.

EXPORTS OF BERGAMOT OIL FROM ITALY[12]

	Kg.	Million Lire
1936	131,500	8
1937	128,700	15
1938	154,500	21
1939	210,600	28
1940	121,100	24
1941	77,000	17
1942	119,900	28
1943	33,700	9
1944	89,900	67
1945	148,400	240
1946	227,200	700
1947	102,000	510
1948	150,000	646

Numerous samples of the bergamot oil produced in southern Italy during the 1947-48 season were analyzed at the Reggio Calabria Experiment Station and the results were summarized by Albricci[13]. In a series of 22 samples of oil representative of various producing areas at different stages of the harvest, the density at

15° varied from 0.8800 to 0.8855 and the optical rotation at the same temperature from + 6.60° to + 24.60°. The proportion of esters, as linalyl acetate, ranged from 33.58% to 44.18% (average 38.77%) and the proportion of free alcohols, as linalool, from 16.29% to 35.27% (average 26.76%). The ester content of the oil produced in January and February, 1948, was generally higher than that of the oil produced in the corresponding months of the previous three years.

Experimental studies on the operation of the "pelatrice" machine which is used for the extraction of bergamot oil were reported by La Face[14]. The studies were concerned with the effects of varying the speed of rotation of the abrading plate and making other changes in operating technique on the efficiency of the machine.

In irradiation experiments made on his own arms, Oppenheim[15] found that bergamot oil sensitizes the skin to the action of grenz rays (very soft X-rays), providing the oil is rubbed in. If applied lightly in a superficial layer it has no sensitizing effect. The reaction of the skin to bergamot oil followed by irradiation is more pronounced with hard grenz rays than with soft rays. Bergamot oil in itself had no irritating action on the author's skin.

Bois de Rose Oil (Linaloe). The Brazilian Ministry of Agriculture estimated the amount of bois de rose oil produced in the States of Amazonas and Pará during 1947 at 193.5 metric tons. This amount represented the output from 47 distilleries. The tremendous drop from the 1946 total of 481.4 tons was due in part to the lack of spring rains which made it difficult to transport the logs by river to the distilleries. Production of the oil in 1948 was placed at 102.6 metric tons and was thus even lower than in the preceding year.

Complete figures on Brazilian exports of bois de rose oil for 1947 are not available but the total for the six months from April through September was 55 metric tons. Exports of the oil during the entire year 1948 amounted to only 60 tons.

The demand for bois de rose oil in the United States diminished as bergamot oil and lavender oil again became available. The wholesale price of bois de rose in New York dropped from $5.00 per lb. in January of 1947 to $3.00 at the end of the year. However, the producers' association in Brazil attempted to resist the trend by fixing their prices at a high level with the result that buyers in the United States purchased relatively little of the oil in 1948. United States imports of bois de rose oil decreased from 205 metric tons valued at $1,367,500 in 1947 to 45 metric tons valued at $220,200 in 1948.

Mexico exported 10.1 metric tons of linaloe oil in 1948 of which 4.8 tons went to the United States. This was only slightly more than ten percent of the amount exported to the United States in 1945.

Naves[16] identified *p*-methyl acetophenone and *l*-tetrahydro-Δ_3-*p*-methyl acetophenone in Brazilian bois de rose oil.

Boronia Ledifolia Oil. Penfold and Morrison[17] examined the volatile oil extracted from some unusual specimens of the shrub *Boronia ledifolia* Gay (*Rutaceae*) which were found near Gralton in New South Wales. The leaves of these plants had an overpowering odor resembling that of oil of rue. Steam distillation of different lots of the leaves and terminal branchlets gave an essential oil having $d^{15}{}_{15}$ 0.835 to 0.852, $\alpha^{20}{}_{D}$ − 1.54° to + 2.88° and $n^{20}{}_{D}$ 1.4266 to 1.4375. Yields varied from 0.12% to 0.46%. Methyl heptyl ketone and methyl nonyl ketone were identified as the principal components of the oil, representing approximately 75% of its weight. There was some evidence to indicate the presence of α-pinene, methyl anthranilate and sesquiterpenes among the minor constituents. The ketones characteristic of this oil are not found in the essential oil from typical plants of *B. ledifolia* Gay nor in the oil from *B. ledifolia* var. *triphylla*.

Boronia Megastigma Concrete. From the concrete essence of *Boronia megastigma* Nees., Naves[18] isolated eugenol and a mixture of dextrorotatory and racemic α-ionone in addition to β-ionone which previously had been identified in the concrete by Penfold (1927).

Bystropogon Mollis Oil. A sample of the essential oil of *Bystropogon mollis* Kth., an aromatic plant indigenous to Argentina, was examined by Fester[19]. *l*-Menthone was found to be the main constituent although the oil had only slight optical activity. This was due to the presence of pulegone and a small amount of *d*-isomenthone. Two unidentified acids, one monobasic and the other dibasic, were isolated from the oil after saponification.

Cabreuva Oil. Naves[20] identified *d*-nerolidol as the principal constituent of cabreuva oil which is distilled in Brazil from the wood of *Myrocarpus fastigiatus* and *M. frondosus* Allem. (*Leguminosae*). Fractionation of a sample of the oil which had $d^{20}{}_{4}$ 0.8875, $n^{20}{}_{D}$ 1.4832 and α_{D} + 8.31° yielded around 80% of *d*-nerolidol and 2.5% of farnesol. Small amounts of other sesquiterpene alcohols including a bisabolol and a cadinol (dihydro derivative m.p. 132–133°) were also present in the oil. Examination of the ozonolysis products indicated that the nerolidol from cabreuva oil contains some of the *alpha* (isopropenyl) isomer whereas nerolidol from petitgrain oil is made up of the *beta* (isopropylidene) form alone.

A fraction of cabreuva oil distilling between 50° and 70° at 3 mm. which was rich in carbonyl compounds was examined by Naves[16]. This fraction, representing 1.2% of the oil, was found to contain tetrahydro-Δ^{3}-*p*-toluic aldehyde, which has a leafy odor, and two ketones: *p*-methyl acetophenone and *l*-tetrahydro-Δ^{3}-*p*-methyl acetophenone. The semicarbazone of the latter melted at 157° and the ketone regenerated from the semicarbazone distilled at 61° at 3.3 mm. and had $[\alpha]^{20}{}_{D}$ − 35.10°.

Calamus Oil. Analytical studies by Sorm and Herout[21] resulted in the isolation of some new odor-bearing constituents and sesquiterpenic compounds from the essential oil of *Acorus calamus* L. (sweet flag). The calamus oil used in the investigation was of Dutch origin and had the following constants: d^{15} 0.9836, $n^{20}{}_{D}$ 1.5075 and $[\alpha]^{18}{}_{D}$ + 6.11° in ethanol.

An unsaturated aldehyde $C_{10}H_{16}O$ having an intense odor of calamus oil was isolated from the lower boiling fractions by means of Girard T reagent. The

aldehyde, which distilled at 115–120° at 9 mm., polymerized easily and gave a semicarbazone m.p. 135°. Fractionation of the non-carbonylic component of the lower boiling fractions of the oil by means of chromatographic adsorption yielded the sesquiterpene calamene and an oil of intense odor having the composition $C_{10}H_{18}O$. This substance, which distilled at 100–112° (bath temperature) at 9 mm., appeared to be a mixture of monocyclic terpene alcohols, possibly terpineols. The $C_{10}H_{18}O$ alcohols, *d*-camphor and the aldehyde $C_{10}H_{16}O$ are the substances responsible for the characteristic odor of calamus oil, although they are present in small amounts.

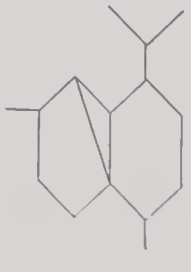
From the intermediate fractions of calamus oil a sesquiterpene ether $C_{15}H_{24}O$ was isolated. This ether, named "acoroxide," contained two double bonds and had b.p. 135–137° at 11.5 mm., d^{20} 0.9179, $n^{20}D$ 1.4970 and $[\alpha]^{20}D$ + 12.1° (± 0.3°) in ethanol. On dehydrogenation of acoroxide no azulene was produced but cadalene was formed.

About half of the oil distilled in the range 113–136° at 0.45 mm. leaving only a small distillation residue. Carbonyl compounds were isolated from the high-boiling portion of the oil by means of Girard T reagent and separated chromatographically. One of the carbonyl constituents was an unsaturated ketone $C_{15}H_{26}O$ which distilled at 96–99° at 0.44 mm. and had a very pleasant odor. This ketone, named "calamone," gave a semicarbazone melting at 185–187°. Another carbonyl compound having m.p. 100–101° and $[\alpha]^{15}D$ + 139.5° in ethanol proved to be a sesquiterpene diketone with the composition $C_{15}H_{24}O_2$. This diketone, named "acorone," contains one double bond and gives a disemicarbazone melting at 205–207° with decomposition. A substance melting at 86° which crystallized from the highest boiling fraction of the oil and from the distillation residue was identified as a molecular compound of acorone and its levorotatory isomeride, m.p. 96–97°, $[\alpha]^{20}D$ −92.4°, which was named "isoacorone." The semicarbazone of isoacorone melted at 196–197° but a disemicarbazone could not be obtained, possibly due to steric hindrance of one of the keto groups. Acorone and isoacorone formed a second molecular compound which melted at 104–105°.

In addition to the ketones and an unidentified aldehyde, the high-boiling fractions of calamus oil contained the dihydric alcohol calameone, $C_{15}H_{26}O_2$, and material with a high methoxyl content which was probably isoasarone, since crystalline asarone failed to separate at low temperatures.

The hydrocarbon calamene was catalytically hydrogenated and absorbed hydrogen equivalent to 1.8 double bonds per molecule. Calamene is therefore probably a mixture of a bicyclic sesquiterpene having two double bonds (*α*-calamene) and a smaller amount of a tricyclic sesquiterpene having one double bond (*β*-calamene). The formation of guaiazulene on dehydrogenation of calamene is evidence of the presence of a tricyclic sesquiterpene for which structure I is suggested.

I



Callitris Glauca Oil. *See* **Cypress Pine Oil.**

Camphor Basil Oil. *See* **Ocimum Canum Oil.**

Camphor Oil. The total production of crude camphor and camphor oil in Japan in 1948 amounted to 3,448 metric tons. This figure represented only about 10% of the annual output before World War II when Formosa was under Japanese control. However, the average monthly production in 1948 was much higher than in the preceding year as shown in Table III. More than 800 camphor factories were in operation in 1948, most of them located in Kyushu. Exports of refined camphor from Japan in 1948 reached a total of 460 metric tons.

Table III.
Average Monthly Production of Camphor Products in Japan[22]
(In Metric Tons)

	Crude Camphor	Camphor Oil	Refined Camphor
1946	42	50	19
1947	85	102	31
1948	132	169	51

The price of crude camphor oil in Japan climbed from 1.50 yen per kg. in June 1910 to 173.00 yen in August 1948. During the same period the price of crude camphor increased from 2.75 yen per kg. to 165.00 yen.

Imports of refined natural camphor into the United States from Japan reached a total of 66,100 lb. valued at $77,700 in 1947 but dropped to only 1,500 lb. in 1948. Imports of Japanese camphor oil in 1948 amounted to 109,300 lb. valued at $25,000 while imports of camphor sassafras oil (the safrole fraction of camphor oil) in 1948 totaled 148,500 lb. valued at $59,000.

United States imports of camphor oil from China increased from 76,450 lb. in 1947 to 106,823 lb. ($34,000) in 1948. Most of the oil was probably produced in Formosa. Imports of refined natural camphor from China in 1948 amounted to 19,600 lb.; in the previous year the import total was insignificant.

Caraway Oil. Khotin[23] reported that the essential oil content of caraway plants is at a maximum from three to five days before the seeds become brown on the central branches. Green seeds give an oil of lower specific gravity and refractive index.

Cassia Oil. Imports of cassia oil from China into the United States in 1947 amounted to 87.3 tons and had a value of $343,400. Imports of the oil in 1948 dropped to 47.4 tons worth $153,800.

Cedarleaf Oil. Bailey[24] investigated the possibility of utilizing the branches of eastern red cedar which are discarded in lumbering operations in the Tennessee Valley for the production of cedarleaf oil. In trial distillations of fresh cedar foliage, carried out at different times of the year, average yields of oil from dominant trees

were as follows: 0.35% in June, 0.43% in October and November and 0.46% in February and March. These figures are somewhat higher than yields reported by other investigators and suppressed trees did not give as high yields. A composite sample of the oil showed d^{15} 0.902, n^{20} 1.4872 and $\alpha + 57.1°$.

Cedarwood Oil. The cedarwood oil industry in the southeastern section of the United States flourished during World War II when there was a strong foreign demand for the oil. However, after the Lend-Lease agreements were terminated some of the older manufacturing plants as well as several new ones in Texas were forced to close down. In the first quarter of 1947 the oil was selling for $0.85–0.95 per lb. but by the end of 1948 the price was down to $0.40.

Kenya exported 45,400 lb. of cedarwood oil valued at £6,750 in 1947. Of this amount 18,600 lb. went to the United Kingdom and 18,000 lb. to the United States. Exports of cedarwood oil from Kenya in 1948 totaled 32,200 lb. (£3,900) and were destined principally for the United Kingdom.

Cinnamon Oil. As shown in Table IV, the quantity of cinnamon oil exported from Ceylon in 1948 was only a little more than half as large as the amount exported in 1938. However, the value was considerably greater due to the rise in price. In 1938 the average price of cinnamon leaf oil was 0.15 rupees per oz. while in 1948 it was Rs. 0.45. In the first quarter of 1948 the price reached Rs. 0.75 per oz. but by the end of the year it had fallen to Rs. 0.24.

Table IV.

Exports of Cinnamon Leaf Oil[a] from Ceylon[25]

	Thousand Oz.	Thousand Rs.
1938	2,786	444
1946	1,239	590
1947	1,134	673
1948	1,484	681

[a] Includes a small amount of cinnamon bark oil.

The cinnamon leaf oil exported from Ceylon in 1948 was distributed as follows: Malaya 474,000 oz., United Kingdom 419,600 oz., United States 180,900 oz. and other countries 400,000 oz. Only 9,000 oz. of cinnamon bark oil was exported in 1948, nearly half of which was shipped to the United States.

The Seychelles Islands exported 57.4 metric tons of cinnamon leaf oil valued at £31,372 in 1947 and 40.1 tons valued at £20,169 in 1948. United States imports of the Seychelles oil totaled 20.3 metric tons in 1947 and 3.1 tons in 1948.

A method for the volumetric determination of cinnamic aldehyde in cinnamon oil, based on the liberation of sulfuric acid in the reaction of the aldehyde

▲ CAMPHOR BASIL PLANT AND FLOWERING TOP.

(See page 40)

Courtesy of Gerda Bruns-Ru

BLOSSOMS OF CAMPHOR BASIL, ENLARGED. ▼

Courtesy of Toichi Kuraoka

CAMPHOR TREES, 25 YEARS OLD, WITH SEEDLINGS IN FOREGROUND.

▼ CAMPHOR TREES, 15 YEARS OLD.

DISTILLING CAMPHOR IN JAPAN. ▼

with hydrazine sulfate, was suggested by Fuchs[26] (cf. Stempel, Chem. Abst. 37: 5196, 1943). The procedure is as follows: Weigh a sample of no more than 0.09-0.11 g. of cinnamon oil in a 100-ml. glass-stoppered Erlenmeyer flask, dissolve in 5 ml. of methanol or 95% ethanol, add 30 ml. of water and titrate with 0.1 *N* NaOH using methyl red as indicator. Add 5 ml. of 0.1 *M* hydrazine sulfate solution and water to bring the weight of the flask contents to 50 g. Allow to stand six hours with occasional shaking. Filter through cotton and titrate a 40-g. aliquot with 0.1 *N* NaOH to the methyl red end point. Titrate 5 ml. of the hydrazine sulfate. One ml. of NaOH is equivalent to 0.0264 g. of cinnamic aldehyde.

Greene's work on the microscopy of essential oils was extended to the oils of cassia, cinnamon bark and cinnamon leaves[27]. A saturated solution of sodium hydroxide in absolute ethanol appeared promising as a reagent for distinguishing between the three oils under the microscope. With this reagent cassia oil gave only an amorphous precipitate while cinnamon bark oil yielded tiny rosettes of crystals and cinnamon leaf oil yielded long, needle-shaped crystals. The addition of a 20% solution of sodium phenate to cassia oil or cinnamon bark oil resulted in the formation of radiating clusters of long needles within ten minutes. These crystals exhibited strong interference colors in polarized light. No crystals formed in cinnamon leaf oil. When treated with phenylhydrazine, cassia oil and cinnamon bark oil (but not the leaf oil) yielded rod-shaped crystals which were colorless in polarized light. The addition of a minute amount of benzidine to either cassia oil or cinnamon bark oil caused the formation of diamond-shaped crystals.

Citronella Oil, Ceylon. Exports of citronella oil from Ceylon were higher in 1948 than in 1947 but lower than in 1946 (see Table V). At the beginning of 1947 the price of citronella oil in Ceylon was approximately 5.85 rupees per lb. as compared with an average price of Rs. 0.73 per lb. for the pre-war year 1938. However, the price could not be maintained at this level and by September it had dropped to Rs. 1.40. During 1948 the overseas demand for the oil was poor but as there were only limited quantities available for shipment the price was fairly stable. The average for the year was Rs. 2.00.

Table V.

Exports of Citronella Oil from Ceylon[25]

	Thousand Lb.	Thousand Rs.
1938	1,601	1,182
1946	1,726	8,908
1947	1,278	3,989
1948	1,568	3,338

The bulk of the citronella oil exported from Ceylon during the first six months of 1948 was destined for the following countries: United Kingdom (268,411 lb.), United States (200,971 lb.), India (78,747 lb.) and Egypt (90,831 lb.). Imports of the oil recorded in the United States for the entire year 1948 totaled 353,070 lb. ($224,000), a considerable decrease as compared with imports of 526,750 lb. ($692,000) in 1947.

Citronella Oil, Java Type. The conflict between Dutch and Republican troops in Indonesia during 1947 provided a further setback to the Java citronella industry which had been disrupted by the Japanese occupation. Exports of citronella oil from Java in 1947 amounted to only 150.2 metric tons as compared with 2,329 tons in 1938. The Netherlands received 124.2 tons and the United States 22.4 tons. Most of the oil shipped in 1947 was old stock which had been in storage for several years and many lots were reported to be low in geraniol and citronellal content. The steam distillation residue in some cases was as high as 14% due to chemical changes in the oil or adulteration with fatty oils. In order to protect the reputation of Java citronella oil, the Department of Commerce in Batavia issued an order in 1948 requiring certificates of analysis from the Laboratory for Chemical Research in Buitenzorg for all large shipments.

The wholesale price of Java-type citronella oil (85/35 grade) in New York was in the vicinity of $4.75 per lb. in January 1947 but in view of the high production in Guatemala the price dropped rapidly. By December it had fallen to $1.20-1.30 per lb. and it remained at this level throughout 1948. The decline in the price of the oil combined with high production costs caused many planters and manufacturers in Java to turn to other enterprises.

In the first half of 1948 sources in Batavia estimated the area of citronella plantings in Java at 30,000 acres as compared with 44,500 acres before the Japanese invasion. Some of the plantations which had been neglected during the war were then being improved by the planting of new grass. Estimates of current production varied between 300 and 600 tons of oil per year.

In order to permit the Java oil to compete more successfully with the citronella oil produced in Central America, the export duty was reduced in May 1948 from 20% to 3%. Exports of citronella oil from Java in 1948 increased to 469 metric tons valued at $676,000 in United States currency. Of the 1948 total, 271 tons went to the United States, 120 tons to the Netherlands and 30 tons to the United Kingdom.

Citronella oil of the Java type is now produced along the northwestern coast of Formosa, where a number of small plantations were started by the Japanese in 1939. United States imports of this oil from China totaled 36,900 lb. in 1947 and 79,800 lb. in 1948.

In spite of the disastrous decline in the price of citronella oil in 1947, production in Guatemala was greatly expanded in 1948. The amount of citronella oil imported into the United States from Guatemala increased from 251,100 lb. (value $679,000) in 1947 to 648,500 lb. (value $638,000) in 1948. Future production of the oil is threatened by a new disease of citronella grass which has appeared in Guatemala. The diseased plants are marked by retarded growth and a reduced content of oil.

United States imports of citronella oil from Honduras totaled 72,400 lb. in 1947 and 65,400 lb. in 1948.

Wit[28] described some of the work done on citronella grass varieties at the Agricultural Experiment Station in Buitenzorg, Java. Citronella plants propagated there from specimens of the grass which had been collected in different parts of Java were analyzed to determine their value for the production of oil. Out of fifty plant "populations", thirty-six fell in a well-defined group characterized by yields (dry basis) of 2.0–2.8% of oil containing 30–40% of citronellal. Most of the remaining populations gave low yields of an oil which was deficient in citronellal while one exceptional group yielded oil with an average citronellal content of 48%. The low-yielding plants in many instances failed to show the morphological traits (red leaf sheath, rough leaf, etc.) listed by Jowitt as characteristic of citronella grass of the "lenabatu" type while high-yielding plants of the "maha pengiri" variety in some cases exhibited these traits.

Three cases of dermatitis resulting from contact with citronella oil were described by Keil[29]. In patch tests on two of the patients, citronellal (1% in acetone) gave a strong vesicular reaction which indicated that this constituent was the main allergen in the oil. The related compounds, hydroxycitronellal and citronellol, produced a milder reaction. Geraniol, which is the most important constituent of citronella oil, elicited no reaction from one patient and a mild papular erythema from the other. Patch tests with these compounds on 14 control subjects gave completely negative results.

In a series of tests of insect repellents for use against the Australian sheep blowfly, Waterhouse[30] found that Ceylon citronella oil was an effective repellent while Java citronella oil was not. The repellent substance in the Ceylon oil was found to be concentrated in one of the lower boiling fractions in which it was mixed with geraniol and citronellal. Neither of these constituents has repellent properties.

Clausena Anisata Oil. The leaves of *Clausena anisata* Hook f., a shrub belonging to the *Rutaceae*, contain an essential oil which is similar in composition to anise oil, according to Meijer[31]. Although Brooks (Philippine J. Sci. 6:341, 1911) reported that oil which he distilled from the same plant in the Philippines consisted mainly of estragole, the leaves of *C. anisata* plants grown at the Agricultural Experiment Station at Buitenzorg, Java, yielded an oil which was rich in anethol. On chilling the oil, anethol readily crystallized out and was identified by oxidation to anisic acid. The oil also contained a small amount of anisaldehyde, identified as the *p*-nitrophenyl hydrazone, m.p. 160°.

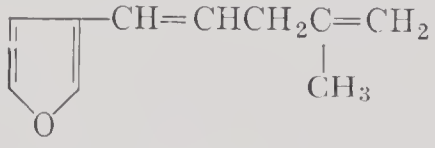
Thirty-one samples of *C. anisata* oil were distilled from 500-g. lots of leaves collected at monthly intervals over a period of a year, seven of the samples being distilled from young leaves. The yields of oil, including cohobated oil, from the old leaves varied from 3.1% to 6.3% (moisture-free basis) and showed no seasonal differences. The yields from young leaves were higher, ranging from 5.1% to 8.2%. Distillation of the oil on a commercial scale would give lower yields than those recorded in the laboratory. The anethol content of

all samples of oil, on the basis of the methoxyl determination, ranged from 74.3% to 89.6% while the average was 84.4%. The physical properties of the oil varied only slightly during the year, the limits for all samples being as follows: $d^{27.5}_4$ 0.9725 to 0.9835, $n^{26}D$ 1.5526 to 1.5594 and αD 0.0° to — 1.76°.

The stems yielded 2.1% of oil (moisture-free basis) which contained 85% of anethol and closely resembled the leaf oil in physical properties.

Meijer considers that anise oil could be produced more cheaply from *C. anisata* than from the fruits of *Illicium verum,* the usual source of commercial anise oil. The most promising method found for propagating *C. anisata* is the grafting of the stock on stems of *C. excavata.* The odor of *C. anisata* oil is somewhat different from that of ordinary anise oil, but there is a possibility that it could be improved by refining the oil.

Clausena Willdenovii Oil. The essential oil of *Clausena Willdenovii* contains a furan derivative called α-clausenan. In view of the reactions of this compound, Narasimha Rao[32] considers that the formula below probably represents its structure.



Clove Oil. The 1946-47 harvest of cloves in Zanzibar and Pemba reached a total of 18,808 (short) tons while the 1947-48 crop amounted to only 5,458 tons. A somewhat larger crop was predicted for the next season. The average price of cloves in Zanzibar for the month of January 1947 was 52 shillings per 100 lb. and for January 1948, 47 shillings. A rise in price occurred during 1948 following the meager harvest and the average price for the month of December 1948 was 73 shillings per 100 lb.

It will be seen from Table VI that India is the leading postwar market for Zanzibar cloves with the Straits Settlements ranking next in volume of imports. The shipment of cloves to Indonesia was suspended during the entire year 1947 as a result of the warfare there.

Table VI.

Exports of Cloves from Zanzibar[33]

	1946 Tons	1947 Tons	1948 Tons
India	9,522	5,615	8,641
Straits Settlements	3,493	999	3,238
Continental Europe	907	159	93
United Kingdom	1,005	281	136
North and South America	866	369	55
Indonesia	120		395
Other countries	1,465	692	561
Total	17,378	8,115	13,122

Exports of clove oil from Zanzibar amounted to 385,905 lb. in 1947 and 333,956 lb. in 1948. Nearly all of the oil was shipped to the United Kingdom although a small quantity was exported to Australia.

The crop of cloves harvested in Madagascar in 1947 amounted to only 2,000 metric tons. In the following year 3,500 tons of cloves were harvested while a portion of the large crop remained unpicked due to a shortage of labor. Exports of cloves from Madagascar during these two years included shipments of the stocks accumulated during the war which brought the total for 1947 to 2,914 metric tons and for the first 11 months of 1948 to 7,239 tons. Exports of Madagascar cloves to the United States totaled 1,336 metric tons in 1947 and 2,463 tons (unofficial estimate) in the first 11 months of 1948. The price of cloves remained steady at $0.06 per lb. f.o.b. Madagascar from July to mid-November of 1948, but then rose sharply, reaching $0.13 per lb. by the end of the year.

Madagascar exported 551.4 metric tons of clove oil in 1947 and 520.9 tons in 1948 as compared with 305.5 tons in 1938. The distribution of clove oil exports is shown in Table VII.

Table VII.
Exports of Clove Oil from Madagascar[34]

	1947 Metric Tons	1948 Metric Tons
France	362.7	394.2
United States	101.9	38.9
United Kingdom	64.7	63.9
Australia	15.1	
Netherlands	0.6	19.7
Other countries	6.4	4.2
Total	551.4	520.9

Caryophyllene, a doubly unsaturated bicyclic sesquiterpene which occurs in clove oil, does not readily undergo autoxidation. However, Treibs[35] found that it absorbed oxygen in the presence of cobalt siccative to give, among other products, a crystalline epoxide $C_{15}H_{24}O$, m.p. 64°. This oxide, formed by the addition of oxygen to the cyclic double bond, could be prepared in high yields by treating caryophyllene with perbenzoic acid or with hydrogen peroxide in the presence of pervanadic acid. Hydrogenation of caryophyllene oxide using a palladium catalyst resulted in saturation of the double bond in the side chain. The dihydrocaryophyllene oxide, $C_{15}H_{26}O$, melted at 67°. Treatment of caryophyllene oxide with hydrogen in the presence of nickel gave a saturated secondary alcohol identical with tetrahydrobetulenol in all respects except its high rotation.

A substance (m.p. 64°) which was isolated from the sesquiterpene fraction of clove oil was shown to be identical with the epoxide of caryophyllene which was prepared synthetically. The constants of the sesquiterpene oxide found in lavender oil by Seidel *et al.* (Helv. Chim. Acta 27:738, 1944) are also identical with those

of caryophyllene oxide. Since caryophyllene is present in both clove and lavender oils, Treibs considered it to be the biological parent substance of the epoxide. However, Naves[36] showed that caryophyllene is not present as such in clove buds. Benzene extraction of cloves yielded an oil containing a small amount of caryophyllene oxide but no caryophyllene while steam treatment of the press cake from the extraction gave an essential oil consisting mainly of caryophyllene. Thus the caryophyllene in clove oil must be formed during steam distillation.

Bartels[37] tested the ability of eugenol and oil of cloves to prevent the growth of various microorganisms on infusion agar. Both eugenol and oil of cloves at a concentration of 0.5% and at pH levels of 6.0, 7.0 and 8.0 inhibited the growth of *B. subtilis*, *Staphylococcus aureus* and *B. coli* but did not affect *B. pyocyaneus*. Eugenol showed strong antibacterial activity against *E. typhi* and *Monilia albicans*.

Convolvulus Pluricaulis Oil. The essential oil distilled from the fresh plants of *Convolvulus pluricaulis* Chois., an herb found in India, was described by Basu and Dandiya[38]. The oil had d^{28} 0.8610, n^{28} 1.4676, acid value 0.79, ester value 11.5 and acetyl value 25.6.

Coprosma Foetidissima. Sutherland[39] isolated methyl mercaptan from the New Zealand plant *Coprosma foetidissima* Forst. by distillation of the leaves with water. This mercaptan is probably the principal substance responsible for the foul odor of the plant.

Costus Oil. At the Forest Research Institute at Dehra Dun, India,[40] costus roots from Garhwal were found to contain nearly 1.4% of essential oil. This figure was low in comparison with costus roots from Kashmir and distillation of the Garhwal roots in the pilot plant yielded only 0.4% of oil. The properties of the oil were as follows: boiling range 50° to 210° at 11 mm., d^{20} 0.939, α + 15.12°, n 1.4982, acid value 8, ester value 43 and ester value after acetylation 75.

The crystalline material which sometimes separates from costus oil on storage was shown by Crabalona[41] to be identical with the lactone $C_{15}H_{18}O_2$ which Ukita isolated from the petroleum ether extract of costus roots (J. Pharm. Soc. Japan 59: 80, 1939). The lactone, which is odorless, occurs in the form of large, prismatic crystals, m.p. 61.5° and $[\alpha]_D$ − 12.53° (alcohol, c = 15). When the lactone is heated above its melting point the viscous melt becomes hard, vitreous and insoluble in organic solvents. The hydroxy acid formed on saponification of the lactone melts at 122°. The lactone is bicyclic and contains three double bonds, yielding a hexahydro derivative $C_{15}H_{24}O_2$ on hydrogenation in the presence of Raney nickel. The saturated lactone is a viscous, odorless oil b.p. 135° at 3 mm., α^{20}_D − 83°, d^{20}_4 1.067 and n^{20}_D 1.5120.

The dihydro derivative of Ukita's lactone was prepared by Naves[42] by reduction with sodium and alcohol. It has an odor of cedar with a rose note and the constants are close to those of the "costus lactone" $C_{15}H_{20}O_2$ isolated from costus oil by Semmler and Feldstein (Ber. 47: 2433, 2687; 1914). The hexahydro lactone $C_{15}H_{21}O_2$ on dehydrogenation with selenium gave a bicyclic dihydro-sesquiterpene and a mixture of guaiazulenes.

Couch Grass Root Oil. Treibs[13] found that the volatile oil from the roots of couch grass (*Agropyrum repens*) consists almost exclusively of a hydrocarbon containing an acetylenic bond. This substance, designated as "agropyrene", is a liquid having b.p. 140-143° at 10 mm., d^{20} 0.9744 and $n^{20}D$ 1.5695. If heated to the boiling point at atmospheric pressure it polymerizes to a soft resin. The odor of agropyrene is described as aromatic, resembling that of methyl chavicol, and extremely tenacious. The identity of the products formed from agropyrene on hydrogenation and oxidation showed it to be an aromatic enyne hydrocarbon having the structure $C_6H_5{\cdot}CH_2CH{=}CHC{\equiv}CCH_3$. Agropyrene is the first representative of this class of hydrocarbons known to occur naturally.

Cymbopogon Densiflorus Oil. (Andropogon Schoenanthus var. Densiflorus, Hack). Thuriaux[44] reported that *Cymbopogon densiflorus*, a plant which is widely distributed throughout the Belgian Congo, appears to be of potential economic value because of the high essential oil content of the blossoms. The plant has been successfully cultivated at the Keyberg Experiment Station and can be harvested twice a year under favorable growing conditions. The fresh inflorescences of *C. densiflorus* yield approximately 2% of essential oil while the stems and leaves yield only 0.1%. The fresh flower oil has a powerful and rather harsh odor which becomes sweeter as the oil ages. The analysis of a few samples of the oil gave the following average values: $d^{20}{}_{20}$ 0.941, αD + 60°, n^{25} 1.489, ester value 12, ester value after acetylation 132; soluble in 1.9 parts of 70% alcohol at 20°. The analytical values change to some extent on aging of the oil.

Cypress Pine Oil. Some of the minor constituents of the wood oil of the cypress pine (*Callitris glauca* R.Br.) were investigated by Neuhaus and Reuter[45]. Saponification of the oil remaining after the removal of *l*-citronellic acid and guaiol yielded an unsaturated acid, $C_{10}H_{14}O_2$, m.p. 186–187°. The saturated acid formed from it on catalytic hydrogenation gave an amide which was identical with tetrahydro-geranamide. The acid $C_{10}H_{14}O_2$ is thus dehydrogeranic acid (3,7-dimethyl-2,4,6-octatrienoic acid). A phenolic substance b.p. 180° at 1.5 mm. which was isolated from the saponification mixture gave the color reactions described by Baker and Smith (1910) as characteristic of a phenol present in *Callitris* wood oils and named by them "callitrol". After hydrogenation the phenolic substance failed to give the color reactions. A fraction b.p. 206–230° at 13.5 mm. which was isolated in the fractionation of crude *l*-citronellic acid gave color reactions different from those of the above phenol. An acetic acid solution of this fraction on treatment with bromine gave a green color turning to blue and on treatment with sulfuric acid gave a lasting red color.

Dacrydium Kirkii Oil. The composition of the leaf oil of *Dacrydium Kirkii*, a pine tree of New Zealand, was investigated by Briggs and Taylor[16]. Two samples of the oil distilled in September and November in yields of approximately 0.1% had the following constants: $d^{25}{}_4$ 0.8798 and 0.8768, $n^{25}D$ 1.4754 and 1.4751, and $[\alpha]^{25}D$ + 38.53° and + 37.30°, respectively. Analysis of the fractions of the oil separated by vacuum distillation showed the presence of the substances listed below, in approximately the percentages given.

Terpenes: *d*-α-pinene, 65%; myrcene, 4%; *d*-limonene, 2%; unidentified, 2%.

Sesquiterpenes: cadinene, 1%; unidentified sesquiterpene b.p. 118-125° at 10 mm., 1.5%.

Diterpene: phyllocladene, 1%.

Alcohols: unidentified sesquiterpene alcohols (a) b.p. 125-134° at 5 mm., 4%; (b) m.p. 42-43°, b.p. 135° at 5 mm. (probably a new sesquiterpene alcohol), a trace; (c) b.p. 100-112° at 0.01 mm., 1.5%.

Ester: *d*-bornyl acetate, 1%.

Ketone: carvone, a trace.

Other compounds, including residue, 17%.

Dillweed Oil. One of the large producers of dill estimates the extent of dill plantings in the United States at 800—1,000 acres and the annual production of dill oil at 15 tons. In Oregon the dill crops of 1947 and 1948 were damaged by a rust disease caused by the fungus *Cercosporella anettii*. Agricultural experts are attempting to find a fungicide which will prevent the disease without leaving a toxic residue on the plants.

Diplolophium Africanum Oil. Naves[1] examined two samples of oil distilled in Tanganyika from the plants and fruit of *Diplolophium africanum* Turcz., an umbellifer. The oil, obtained in an average yield of 1%, had an aromatic, camphor-like odor. One sample had d^{20} 0.9486, $n^{20}D$ 1.4880 and αD + 16.80° while the other showed d^{20} 0.9578, $n^{20}D$ 1.4907 and αD + 16.44°. The ketone content of the first sample was 58.4% and of the second, 63.8%. The ketone $C_{10}H_{16}O$ which is the main constituent of the oil is dextrorotatory and gives a semicarbazone m.p. 204-205°.

Eucalyptus Oils. Australian exports of eucalyptus oil for the fiscal year ending June 30, 1947, amounted to 840 tons and had a value of £Aus 408,000. In 1947-48 the total dropped to 741 tons valued at £Aus 324,000.

Table VIII.

Exports of Eucalyptus Oil from Australia[47], 1946-47[a]

	Lb.	£ Aus
United Kingdom	533,914	133,277
Canada	27,914	8,100
India	45,159	9,448
Malaya	18,046	12,103
New Zealand	65,767	11,723
Union of South Africa	45,540	12,821
France	123,119	32,130
United States	691,085	162,971
Other countries	99,917	25,578
Total	1,680,461	408,151

[a] Year ending June 30.

LEAVES, BLOSSOMS AND FRUIT OF AUSTRALIAN EUCALYPTS.

Eucalyptus Australiana, R.T.B.

Courtesy of A. R. Penfold

Eucalyptus Citriodora Hook.

Eucalyptus Dives, Sch.

Eucalyptus Polybractea, R.T.B.

Ralph[18] investigated the minor constituents of the essential oils of *Eucalyptus dives* var. C and *E. australiana*. These oils are very similar in composition, both containing a large proportion of cineol and some α-terpineol and citral. Acids and esters which are present in the crude oils remain for the most part in the still residue on rectification. A typical sample of the crude oil of *E. dives* var. C had an ester value of 11.9 corresponding to 4.4% of methyl cinnamate and an acid value of 2.1, equivalent to 0.7% of geranic acid. Eudesmol crystallized from the highest-boiling fractions of the oil. The still residue from the oil of *E. australiana* contained approximately 2% of methyl cinnamate and 0.2% of geranic acid based on the crude oil, in addition to some free cinnamic acid. Eudesmol was not found in this oil. A hydrocarbon having constants similar to those of aromadendrene and an unidentified sesquiterpene alcohol were isolated from the high boiling fractions.

Eucalyptus Campaspe Oil. The oil of *Eucalyptus campaspe*, obtained in a yield of 1.22%, was examined by Watson and Gardner[19]. The properties of the oil were as follows: d^{15} 0.9225, n^{20} 1.465, α + 7.15°; soluble in 1 volume of 80% alcohol or 10 volumes of 70% alcohol. *E. campaspe* oil was found to contain 64% cineol, 7.2% geraniol, 1.8% aldehydes as $C_{10}H_{14}O$ and 1.2% esters as $C_{12}H_{20}O_2$. It gave the color reactions characteristic of aromadendrene.

Eucalyptus Citriodora Oil. In a study of five specimens of *Eucalyptus citriodora*, Penfold *et al.*[50] compared the oil yields from leaves of different stages of maturity picked from the same tree. It was found that the young mature leaves gave higher yields of oil than the old leaves while the latter gave higher yields than the immature "tips". The citronellal content of the oil was at a maximum in the young full-grown leaves. This work emphasizes the importance of selecting foliage samples of approximately the same degree of maturity for the purpose of comparing the oil yields of individual trees. Storage tests showed that *E. citriodora* foliage collected for use in analytical studies could be stored for two weeks without appreciable loss of oil or decrease in the citronellal content of the oil.

The discovery of a physiological form of *Eucalyptus citriodora* Hooker which yields an essential oil containing less than 10% of citronellal was reported by Penfold and Morrison[51]. Citronellol and its esters are the main constituents of this atypical oil which has a higher specific gravity than the usual oil of *E. citriodora*.

Eucalyptus Cneorifolia Oil. The seasonal variations in the composition of the essential oil of *Eucalyptus cneorifolia* were studied by Berry[52] in a continuation of earlier work on the subject (J. Chem. Soc. 1937, 1443). The occurrence of this species of eucalypt is confined almost entirely to Kangaroo Island off the southern coast of Australia. The branches of the shrubs begin to put out new growth in September (spring in Australia) and the highest yields of oil are realized when the young leaves are nearly full grown. This stage, known as the flush, occurs in November and early December and usually lasts for three or four weeks.

Distillations of the tips of the branches of *E. cneorifolia* were carried out every month from October 1937 through January 1939 and the oils were analyzed for cineol, cryptone, aldehydes, cymene and terpenes. Other samples of oil dis-

tilled at the peak of the flush period in 1939, 1941, and 1943 were also examined. Yields during the flush were usually in the range 2.0—2.6% in comparison with yields of 1.0—1.7% during the rest of the year.

With the start of the new growth in September the density of the oil, which remained at 0.92 during the winter months, began to decrease and reached a minimum of approximately 0.88 at the peak of the flush period. At the same time the negative optical rotation of the oil was increasing. These changes were due to the formation of terpenes in the new leaves, the terpene fraction of the oil consisting of *l*-α-phellandrene, *l*-β-phellandrene and a small amount of dipentene. The oil distilled from rapidly growing tips contained 42—57% of terpenes and 30—40% of cineol but as soon as the leaves had matured the terpene content of the oil declined rapidly. During the period from February to August the oil failed to give a positive test for phellandrene and consisted mainly of cineol (55—62%).

Cryptone was extracted from the oil by shaking it with sodium sulfite solution with continuous neutralization of the sodium hydroxide liberated in the reaction. As the ketone reacted with more than the equivalent amount of sodium sulfite in the presence of an excess of the reagent, it was impossible to determine cryptone on the basis of the amount of sulfuric acid required for neutralization. Consequently the cryptone was liberated from the sulfite solution by means of concentrated alkali and then weighed. The cryptone content of *E. cneorifolia* oil remained at 7—10% from January until the new shoots started in September and then dropped to only 1—3% at the peak of the flush period.

The aldehyde content of the oil was also at a minimum during the flush period. In November and early December the aldehydes (cuminal and phellandral) made up less than 5% of the oil as compared with 6—11% from January to August.

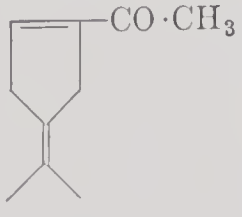
The work done in 1937 (*loc. cit.*) indicated that the cymene content of *E. cneorifolia* oil declined during the winter. However, it was not realized at that time that the determinations of cymene were decidedly unreliable because of the presence of a large amount of cineol in the cymene fraction. Treatment with resorcinol solution was used to separate cineol from the hydrocarbons (terpenes and cymene) but the separation was incomplete. When the hydrocarbon fraction was treated with potassium permanganate to destroy the terpenes, the cineol as well as the cymene remained unaffected and distilled with it when the mixture was subjected to steam distillation. In the later work a correction was made for the cineol in the cymene fraction and the results, although only approximations, permitted a more accurate comparison of the different samples in the series. The cymene content of the oil appeared to be lowest (less than 4%) during the state of rapid leaf growth and fluctuated between 4% and 10% during the remainder of the year.

The fact that the proportion of carbonyl compounds in *E. cneorifolia* oil begins to rise directly after the flush period while the proportion of terpenes is decreasing indicates that cryptone and the aldehydes are formed at the expense

of the terpenes. It is probable that phellandral and cryptone are formed by the oxidation of β-phellandrene and that cuminal is derived from α-phellandrene. The cymene present in *E. cneorifolia* oil may be an oxidation product of α-phellandrene and possibly undergoes partial conversion to cuminal.

Eucalyptus Globulus Oil. Schmidt[53] isolated several carbonyl compounds including a new ketone from the high-boiling fractions of *Eucalyptus globulus* oil of Spanish origin. The crude oil contains approximately 60% cineol, 5—10% free and esterified alcohols and 5% of carbonyl compounds, the remainder of the oil consisting of pinene, aromadendrene, and other hydrocarbons. The last runnings from the commercial rectification of Spanish eucalyptus oil were fractionated and carbonyl compounds were extracted from each fraction by means of sodium sulfite-bicarbonate solution. Treatment of the sulfite compounds with sodium carbonate followed by steam distillation yielded dextrorotatory mixtures of carbonyl compounds. The following components were identified: *d*- and *d,l*-myrtenal, *l*- and *d,l*-carvone, *d*-verbenone and dihydrocuminaldehyde. Tetrahydrocuminaldehydes were apparently present but were difficult to isolate.

Decomposition of the remaining sulfite compounds with sodium hydroxide and steam distillation of the products yielded a highly levorotatory substance which was identified as *l*-pinocarvone and an optically inactive ketone, b.p. 225° at 738 mm., which had a pleasing, anise-like odor. The ketone had d^{15} 0.955 and $n^{20}D$ 1.4911, solidified at — 0.2° and gave a semicarbazone m.p. 202°. It had the molecular formula $C_{10}H_{14}O$ and contained two ethylenic bonds, one in conjugation with the carbonyl group. On hydrogenation with palladium the compound was converted to 3-isopropyl-1-acetyl cyclopentane, semicarbazone m.p. 163°. The extracyclic position of the second double bond was shown by reduction of the ketone to pinolone. The substance $C_{10}H_{14}O$ was accordingly identified as 3-isopropylidene-1-acetyl-5-cyclopentene (I).



I

Pinocarvone may be a constituent of other eucalyptus oils, especially those which contain a high proportion of cineol and phellandrene. On aging they deposit an insoluble white substance which is probably polymerized pinocarvone.

Eucalyptus Kochii Oil. Three samples of oil from *Eucalyptus kochii* were analyzed by Watson and Gardner[49]. Average values for the constants were as follows: *d* 0.923, *n* 1.460, α + 2.1°; soluble in 2 volumes 70% alcohol. The samples contained 85%, 87.5% and 92% of cineol, respectively.

Eugenia Haitiensis Oil. The essential oil from the leaves of *Eugenia Haitiensis* Krug. & Urb. is used in Haiti as an insecticide. The constituent responsible for

the insecticidal activity of the oil was identified by Jacobson and Haller[54] as 1,8-cineol (eucalyptol). The cineol fraction constituted 86% of the sample of oil which they examined.

Fern Oil. ***See*** **Asplenium Lamprophyllum** ***and*** **Paesia Scaberula Oil.**

Ferula Foliosa Oil. Kir'yalov[55] investigated the essential oil of the fruit of *Ferula foliosa* Lipsky. Steam distillation of the crushed fruit for 5–6 hours yielded 1.93% of oil having d^{20}_{20} 0.9161, $n^{20}D$ 1.4905, $\alpha^{16}D$ + 8.93°, ester value 11.4 and ester value after acetylation 39.1. The oil contained 35–40% of *d*-β-pinene, $\alpha^{16}D$ + 21.02°. A higher boiling fraction which distilled at 80–142° at 6–8 mm. and made up 55% of the oil appeared to consist of oxygenated sesquiterpene compounds. It contained no esters or substances having active hydrogen and only a trace of carbonyl compounds. The fraction gave the blue color with bromine in chloroform which indicates the presence of substances having the azulene skeleton and when heated with selenium gave cadalene and an azulene. Additional oil was obtained from the fruit by prolonged steam distillation or extraction with acetone, bringing the total yield of oil to 4.5%. This material yielded two fractions, one b.p. 150–155° at 13–15 mm., d^{20}_{20} 0.9691, $n^{19.5}D$ 1.5020 and the other b.p. 155–178° at 13–15 mm., d^{20}_{20} 0.9860 and $n^{19.5}D$ 1.5050. These fractions contained oxygen and on being heated with selenium they gave traces of azulene and much cadalene. Treatment of the lower boiling fraction with hydriodic acid gave an iodide m.p. 122–123°. The occurrence of the dextrorotatory form of β-pinene in the oil of *Ferula foliosa* is interesting as it has been found in only a few other essential oils, one of these being the fruit oil of *F. galbaniflua*.

Ferula Jaeschkeana Oil. Kir'yalov[56] reported that the α-pinene which occurs in the essential oil of the fruits of *Ferula Jaeschkeana* Vatke is optically active while the α-pinene in the essential oil of the roots is inactive. The α-pinene from the fruit oil gave a pinonic acid the semicarbazone of which melted at 203–204° while the α-pinene from the root oil gave a crystalline pinonic acid of m.p. 103–104°, the semicarbazone of which melted at 206–207°. A new constituent, camphene, was found in both the fruit and root oils in the proportion of 2–5%.

Feverfew Oil. Kidder[57] reported that steam distillation of feverfew plants (*Matriacaria parthenium* L.) gave 0.25% of an essential oil having an odor similar to that of rosemary oil but more powerful. On standing, the oil deposited crystals of *l*-borneol.

Fir Needle Oil. Preliminary studies relating to the development of a fir tree oil industry in Komi in the U.S.S.R. were described by Grekhmev[58]. Steam distillation of fir twigs picked in June gave an average yield of 2.7% of oil (on a dry basis) while twigs picked in July yielded 3.5% of oil. It was found that the branches could be stored for as long as thirty days without loss of oil. Analysis of two samples of fir needle oil showed a bornyl acetate content of 43%.

Garlic Oil. Stoll and Seebeck[59] isolated alliin, the parent substance of garlic oil, by extraction of garlic with methanol at low temperatures. After purification, alliin crystallized in long, colorless needles, m.p. 163–165°, $[\alpha]^{21}D$ + 62.8° in water.

Analytical studies showed the substance to be a new amino acid having the structure $CH_2{:}CHCH_2\overset{O}{\overset{\|}{S}}CH_2\overset{NH_2}{\overset{|}{C}}HCOOH.\frac{1}{2}H_2O$. Under the influence of the enzymes in garlic, alliin is converted to allicin, $CH_2{:}CHCH_2\overset{O}{\overset{\|}{S}}SCH_2CH{:}CH_2$, which is the antibacterial principle of garlic. The loss of the oxygen atom transforms allicin into diallyl disulfide, the main constituent of garlic oil. Conversely, diallyl disulfide may be oxidized to allicin by treatment with hydrogen peroxide in acetic acid.

Geranium Oil. The geranium crops harvested on Réunion Island during 1947 were normal. However, only 48 metric tons of geranium oil were exported in 1947 as compared with 106 metric tons in the preceding year. The 1947 exports, which had a value of 77.2 million francs, were distributed as follows: 32.2 tons to France, 1.5 tons to Madagascar, 3.6 tons to the United Kingdom and 10.7 tons to the United States. A severe cyclone struck Réunion in January of 1948, destroying a large portion of the crop for that year. Following this disaster the price of the oil jumped from 1500 francs to 2800 francs per kg. within four months. Exports of geranium oil from Réunion during 1948 totaled 57.8 metric tons.

Gingergrass Oil. ***See*** **Palmarosa Oil.**

Goldenrod Oil. The cultivation of sweet goldenrod, *Solidago odora* Ait., as an essential oil crop was undertaken at the Texas Engineering Experiment Station[60]. It was found that goldenrod could be propagated successfully by dividing the clumps after the start of growth in the spring. The plants require little attention while growing and are disease resistant. The oil yield is highest when the plants are in full bloom in late September and is diminished if the plants are allowed to dry out after cutting. A typical lot of goldenrod gave a 2.1% yield of oil having d^{30} 0.939, $[\alpha]_D$ + 14° and n 1.5095 and soluble in 6.6 volumes of 80% alcohol. *d*-Limonene and *l*-α-pinene were identified in the oil of sweet goldenrod, the composition of which is approximately as follows: methyl chavicol 75%, *d*-limonene 15%, *l*-borneol 3%, esters as bornyl acetate 3% and *l*-α-pinene 0.2%. The experimental work indicates that the oil, which resembles anise oil in odor and flavor, could be produced easily and economically on a commercial scale.

Grapefruit Blossom and Leaf Oils. Sabetay and Halasz[61] described the essential oils prepared from the blossoms and leaves of grapefruit trees. The blossom oil (yield 0.03%) had an odor resembling that of neroli but with more of a honey and rose character. The oil obtained from the leaves and branches (yield 0.4%) had a more insipid odor than that of ordinary petitgrain. The constants of the two oils are shown below.

	Blossom Oil	Leaf Oil
$n^{20}D$	1.4761	1.4721
d^{15}	0.8694	0.8607
α	+ 33.20°	+ 55.20°
Acid value	1.7	2.2
Ester value	10.3	10.6
Aldehydes (as aldehyde C_{10})	4.94%	2.81%
Methoxyl (as methyl anthranilate)	1.6%	0.34%

Grapefruit Oil. Results of the examination of two samples of grapefruit oil from Palestine were reported by Islip and Major[62] of the British Imperial Institute. The constants of the two oils, one of which was received in 1948 and the other in 1947, are shown below.

	1948 Oil	1947 Oil
d^{20}_{20}	0.8574	0.8697
$\alpha^{20}D$	+ 93.02°	+ 89.60°
$n^{20}D$	1.4770	1.4790
Aldehydes, as octyl and decyl	1.00% (w/w)	0.77% (w/w)
Non-volatile matter	7.9% (w/w)	9.9% (w/w)

The 1948 oil, which is described as having a well-defined fragrant odor and a good but rather weak flavor, shows physical constants in close agreement with the figures given by Nelson and Mottern for Florida grapefruit oils (Ind. Eng. Chem. 26: 634, 1934). The aldehyde content of 1.0% is somewhat low in comparison with that of the Florida oils (1.6—1.74%). However, the fact that it was determined by a different analytical method must be taken into consideration. The earlier sample of Palestinian grapefruit oil is deficient in aldehydes and the physical constants fall outside the limits given by Nelson and Mottern.

Kesterson and McDuff[63] analyzed six samples of coldpressed grapefruit oil produced in Florida during the 1947-48 season. The analytical values varied between the following limits: d^{25}_{25} 0.8508 to 0.8532, $n^{20}D$ 1.4746 to 1.4761, $\alpha^{25}D$ + 91.19° to + 92.96°, aldehyde content 1.49% to 1.67%, ester content 2.11% to 4.20% and evaporation residue 6.02% to 8.02%. Nine samples of vacuum steam-distilled grapefruit oil obtained in the de-oiling of grapefruit juice for canning showed d^{25}_{25} 0.8415 to 0.8539, $n^{20}D$ 1.4714 to 1.4746, $\alpha^{25}D$ + 91.50° to + 96.5° and evaporation residue 0.19% to 3.66%. The aldehyde content of the samples ranged from 2.3% to 4.06% and was thus much higher than that of the coldpressed grapefruit oil while the ester content (0.08% to 2.52%) was lower. Similar differences between the vacuum distilled and coldpressed oils were found in the case of orange oil (see page 43).

Helichrysum Concrete. *d*-α-Pinene was identified in the concrete extract from everlasting plants (*Helichrysum angustifolium* DC) by Crabalona and Teisseire[64]. The portion of the concrete which was volatile with steam yielded a fraction b.p. 33—35° at 8 mm., $\alpha^{20}D$ +43°40′, which was identified as α-pinene by the formation of pinonic acid on oxidation with potassium permanganate. *d*-α-Pinene constitutes from 2% to 10% of the steam-volatile portion of the concrete.

Horseradish Oil. Stoll and Seebeck[65] demonstrated that the parent substance of the allyl isothiocyanate in horseradish oil is identical with the glycoside sinigrin which occurs in mustard seed, thus confirming the opinion expressed by Gadamer (Arch. Pharm. 235: 577, 1897). The crystalline glycoside isolated from horseradish root melted at 127—128° and had the molecular formula $C_{10}H_{16}O_9NS_2K$. When treated with the enzyme myrosinase from white mustard seed it gave off the intense, sharp odor of horseradish.

Hyssop Oil. Delépine and Badoche[66] reported that the *l*-pinocamphone which occurs in hyssop oil is largely the *cis* form (isopinocamphone). The pinocamphone

fraction of hyssop oil (b.p. 75–90° at 11 mm.) gave a semicarbazone of m.p. 281° and $[\alpha]_D$ + 108° (in acetic acid, c 0.03). *d*-Isopinocamphone prepared synthetically by the oxidation of *l*-isopinocampheol gave a semicarbazone m.p. 281–2° and $[\alpha]_D$ – 108° (in acetic acid, c 0.03). [Schmidt (Chem. Abst. 40: 5413, 1946) gives 219–220° as the melting point of the semicarbazone of *d*- or *l*-isopinocamphone.]

Jasmine. La Face[67] determined the variations in yield and quality of jasmine absolute from the blossoms of *Jasminum grandiflorum* picked in Reggio Calabria at different stages of the blooming season and at different hours of the day. The jasmine harvest lasts from the end of June through the month of October and is thus longer than it is in southern France. La Face recorded the highest yield of blossoms for the season in August and the yield of concrete obtained by petroleum ether extraction was also at a maximum (3.3%) in August. The concrete (m.p. 50°) yielded 54.8% of absolute or slightly more than the concretes prepared in July or in September. The absolute contained 39.6% esters as benzyl acetate and 28.9% of substances volatile with ethylene glycol. The jasmine absolute produced in November had a low density and low ester content, indicating that the harvest season should not be prolonged beyond the end of October.

Jasmine flowers open in the late afternoon and are usually picked early the next morning when the perfume content is highest. La Face found that newly opened blossoms which had been picked as buds between 5 and 7 P.M. yielded a concrete having an herbaceous odor. The concrete gave a 51% yield of absolute which had a density of 0.9542, an optical rotation of + 3.12° and ester content of 31.9% as benzyl acetate. Blossoms picked in the early morning (5–7 A.M.) gave a concrete with a floral odor from which was obtained a 54% yield of absolute having *d* 0.9758, α + 0.3° and an ester content of 40.9% as benzyl acetate.

Juniper Wood Oil. A volatile oil was isolated by Kurth and Lackey[68] from the root wood of the Sierra juniper (*Juniperus occidentalis* Hooker) by steam distillation of the acetone extract. Fractionation of the oil yielded 49.3% of cedrene and a cedrol fraction. The acetone extract of wood from the bole of the tree on steam distillation gave crystalline cedrol in yields of 0.9–1.25% of the weight of the wood.

Juniperus Macropoda Oil. The volatile oil obtained from the berries of *Juniperus macropoda* Boiss., an Indian species of juniper, was examined at the Forest Research Institute[10] in Dehra Dun. Samples of *J. macropoda* berries from three different localities in Baluchistan gave oil yields of 1.55%, 1.10% and 2.04%, respectively. Constants of the three samples of oil were as follows: d^{20} 0.8379, 0.8355 and 0.8343; n^{20} 1.4674, 1.4680 and 1.4610; α^{20} + 12.56°, + 10.69° and + 18.18°. The oil contained 60–70% pinene, 30–35% oxygenated compounds and a small amount of cadinene. *J. macropoda* oil was used in India during the war as a replacement for juniper oil from imported berries of *J. communis* L. However, to make the oil acceptable for use in gin under ordinary circumstances it would be necessary to remove most of the pinene which gives the oil an odor of turpentine.

Labrador Tea Oil. Kir'yalov[69] isolated from the essential oil of Labrador tea (*Ledum palustre*) an acyclic terpene which was probably myrcene and a new

sesquiterpene alcohol. The oil was obtained from the fresh leaves in yields of 0.7—1.6% and had d^{20}_{20} 0.9085, $n^{20}D$ 1.4882 and $\alpha^{18.5}D$ — 12.5°. The sesquiterpene alcohol ledol, $C_{15}H_{26}O$, crystallized from the oil on cooling and was removed by filtration. The filtrate, on distillation, yielded a hydrocarbon fraction b.p. 68—73° at 5 mm. having the composition $C_{10}H_{16}$. This substance appeared to be myrcene as it polymerized readily and yielded dihydromyrcene dibromide on sodium-alcohol reduction and treatment with bromine. A higher boiling fraction consisting mainly of alcohols was treated with potassium permanganate to remove easily oxidizable substances. Steam distillation of the unreacted portion yielded a saturated alcohol $C_{15}H_{26}O$ having b.p. 131—133° at 3 mm., d^{20}_{20} 0.9646, $n^{20}D$ 1.4910 and $\alpha^{17}D$ — 15.5°. This new alcohol, which was named "palustrol", was easily dehydrated and when heated with 90% formic acid yielded a diene $C_{15}H_{24}$, b.p. 100—106° at 3 mm. and d^{20}_{20} 0.9058. Treatment of the diene with selenium gave an azulene, the picrate of which melted at 118—119°. These reactions of palustrol indicate that it is a tertiary tricyclic sesquiterpene alcohol closely related to ledol.

Lavandin Oil. ***See also*** **Lavender Oil.** On the basis of analyses of many samples of lavandin oil, Igolen[70] gives the following limits for analytical values:

d^{15}	0.888 to 0.904
$\alpha^{20}D$	0°50′ to — 7°
$n^{20}D$	1.4576 to 1.4660
Ester value	33 to 100
Ester content as linalyl acetate	11% to 35%
Solubility	Soluble in 1 to 4 volumes 70% alcohol, occasionally with slight turbidity on dilution; usually soluble in 5 volumes and more 65% alcohol.

The same author lists the proportions of the more important constituents of lavandin oil as follows:

Linalyl acetate	20%
Linalool	43 — 44%
d-Camphor	6.5%
Cineol	4.8%
d-α-Pinene	1.5%
Borneol	3 — 5%
3-Octanone	1.6%
Sesquiterpene alcohols	1%
d-*n*-Octanol	0.6 — 1%
Hexyl acetate	0.8 — 0.9%
n-Hexanol	0.5%
Bisabolene	0.5%
Geraniol	0.3%
Octyl acetate	0.27%

Lavandula Stoechas Oil. Two samples of volatile oil from *Lavandula Stoechas* L. collected at Coimbra and Azeitão in Portugal were examined by Costa and Cardoso do Vale[71]. The physical and chemical properties are shown in Table IX in comparison with the properties of the oil of *L. pedunculata* Cav.

The authors believe that several samples of "*L. Stoechas*" oil described in the literature were incorrectly identified since the analytical values correspond more closely to those of *L. pedunculata* oil. The latter contains pinene, camphene, cineol, fenchone, camphor and borneol. Analysis of *L. Stoechas* oil showed the

Courtesy of A. Fernandes Costa

LAVANDULA PEDUNCULATA.

TABLE IX.

PROPERTIES OF L. STOECHAS AND L. PEDUNCULATA OILS OF PORTUGAL

	L. Stoechas Oil		*L. Pedunculata* Oil[a]		
	Coimbra	Azeitão	Coja (1936)	Coja (1939)	Freixo de Espada à Cinta
d^{15}_4	0.9316 (20°)	0.9381	0.9354	0.9371	0.9197
α^D	− 14°43′	− 27°	+ 19°57′	+ 17°22′	.....
n_D	1.4812	1.4715	1.4691	1.4697	
Acid value	3.7		0.66	1.36	2.58
Ester value	114	79	9.37	19.1	12.93
Ester value after acetylation	185	112	19.7	62.5	19.9
Ketones, %[b]	0	7	59.17		50.77
Camphor, %[c]		1.22	19.25	15.95	40.0[d]
Cineol, %	17	28	.....	.....	.....

a) Noticias farm. 5: 438, 1939
b) By hydrogenation
c) Aschan method
d) Oil solidified due to high camphor content

presence of cineol, fenchone, camphor and a carbonyl compound giving an oxime m.p. 93–95°. Pinene was not found and the presence of borneol was doubtful. The oil contains a highly levorotatory alcohol and gives a color test for silvestrene, in both these respects differing from *L. pedunculata* oil. *L. Stoechas* oil may also be distinguished by its *levo* optical rotation, relatively high content of esters and alcohols and low content of camphor. Samples of *L. Stoechas* oil examined at one factory in Portugal during the 1941 season showed limiting analytical values as follows: d^{15} 0.939–0.944, α_D greater than – 25°, $n^{20}D$ 1.469–1.475, acid value above 3, saponification value above 80, soluble in 2–3 volumes of 70% alcohol and in less than 1 volume of 80% alcohol.

Lavender Oil. The lavender harvest in southern France in 1948 was not large as it was confined almost entirely to the cultivated lavender crop. The gathering of wild lavender in the mountains was not considered worth while because of the scarcity and high cost of labor. The amount of lavender oil produced in 1948 has been estimated at 60 metric tons. A large crop of lavandin was harvested in that year and production of the oil amounted to 200 metric tons or 20 tons more than in 1947.

Statistics on total French exports of lavender and lavandin oils are not available. Imports of these oils into the United States dropped from 345,300 lb. (value $2,147,000) in 1946 to 165,600 lb. (value $956,700) in 1947. United States imports rose again in 1948, reaching a total of 335,700 lb. with a declared value of $567,800. A comparison of the value of the lavender and lavandin imports for 1946 and 1948 provides striking evidence of the decline in price of these oils, even if allowance is made for a possibly higher proportion of lavandin in the 1948 imports. The wholesale price of lavender oil in New York ranged from $10 to $12 per lb. (40/42 grade) during the first quarter of 1947 but was down to $6–7.25 per lb. in the last quarter of 1947. At the same time the price of lavandin oil dropped from around $3.80 per lb. to $2.00. In the last quarter of 1948, lavender oil was selling at $3–4 per lb. and lavandin oil at $1.30. The rapid decline in price was due to the weakness of the franc as well as to the large surplus of the oils on the French market.

Lavender oil of exceptionally fine quality is now produced in southern France in the Quercy region, Department of Lot, according to Caujolle *et al*[72]. Plantings of lavender were started in the country between the Dordogne and Lot rivers some ten years ago from stock obtained in the Pyrenees and Alps. The lavender fields are located on arid slopes and plateaus at altitudes of only 200 to 300 meters. The oil is distilled at cooperative distilleries at Cahors and Martel, production at Cahors amounting to 800 liters in 1946. The Quercy lavender oil has d^{15} 0.8868 to 0.8927, α^{20} – 8°16′ to – 10°45′ and is soluble in 2.5 to 5.0 volumes of 70% alcohol at 15°. Samples of the oil examined by the authors contained from 45.3% to 53.6% esters as linalyl acetate, from 27.1% to 34.1% of free alcohols and approximately 1.8% of ketones. The odor of the oil has a fruity quality due to the presence of linalyl butyrate and other fatty acid esters of linalool.

Bénezet[73] reported the identification of methyl heptenone, carvone, thujone and pinocamphone in French lavender oil. A study of nitrogen compounds in the oil showed the presence of two cyclic bases which were not identified and a trace of methyl anthranilate.

Experiments conducted by La Face[74] indicate that the harvesting of cultivated lavender should be started when the blossoms begin to wither and may be continued for ten days before the oil yield declines. Delays in distilling lavender after it was cut had an adverse effect on the yield and quality of the oil. Raising of the steam pressure from three atmospheres to five atmospheres shortened the time required for distillation and resulted in a higher yield of oil. The ester content of the oil also increased as a result of the increase in pressure.

Ledum Palustre Oil. ***See*** **Labrador Tea Oil.**

Lemon Oil. The amount of lemon oil produced in Sicily from the 1946–47 crop was estimated at 300 metric tons and approximately the same quantity was expected from the 1947–48 crop. The countries making the largest purchases of lemon oil from Italy in 1947 were the United Kingdom, the United States, France, the Netherlands and Switzerland. Total exports of lemon oil in 1947 amounted to 285,000 kg. and in 1948 to 297,800 kg. (value 936,905,000 lire). United States imports of Italian lemon oil in 1948 totaled 213,850 lb. or more than four times the 1947 figure. In January of 1948 the price of lemon oil was L 2,500 to L 3,000 per kg. but it rose in March to L 3,700–4,000, remaining at this level until November. With the approach of the new season the price then dropped to L 3,000–3,200 per kg.

The quantity of lemon oil produced in California in 1947 was estimated at 100 to 500 tons. The wholesale price of the California oil remained steady at around $3.35 per lb. during 1947 and the first half of 1948 but was reduced in August to $2.65 per lb.

Lemongrass Oil. Production of lemongrass oil in southern India during the 1947–48 season (July–January) was estimated at 350 to 400 long tons as compared with 550 long tons in 1946–47. In the 1948–49 season lemongrass acreage was reduced by an estimated 5,000 acres and the quantity of oil produced probably did not exceed 300 long tons. The downward trend in production was the result of a diminishing demand for the oil abroad and the consequent drop in prices. The export price of Indian lemongrass oil was about $3.50 per lb. c. & f. New York in December 1946 but it dropped steadily during the next eight months, reaching $1.10 in August and September 1947. The market then became firmer and the oil was selling for $1.50 per lb. in December. In the third quarter of 1948 the price was again down to the low reached in the preceding year but it was somewhat higher in the last quarter.

Exports of lemongrass oil from south India in the year ending March 31, 1948 totaled 732,000 lb. with a value of 3,654,000 rupees. This was a considerable drop from the total of 1,154,000 lb. recorded in the preceding year. The United States received 319,900 lb. and the United Kingdom 179,600 lb. of the lemongrass oil exported in 1947–48. In the following year total exports of the oil amounted

to 760,900 lb. (2,406,000 rupees) of which 305,800 lb. went to the United States and 193,000 lb. to the United Kingdom.

While United States imports of lemongrass oil from India were declining, imports of the oil from Guatemala were on the increase. The United States received from Guatemala 109,400 lb. of lemongrass oil, worth $175,000, in 1946; 130,000 lb., worth $236,500, in 1947; and 142,300 lb. ($163,400) in 1948. An Association of Citronella and Lemongrass Growers was organized in Guatemala in 1948 to handle the sale and export of all essential oils produced in the country. The lots of oil which are brought to the warehouse of the Association by different producers are purified and blended so that foreign buyers are now assured of oils of uniform quality.

Haiti is producing increasingly large amounts of lemongrass oil for the American market. United States imports of the Haitian oil totaled 18.8 tons in 1947 and 40.3 tons in 1948.

Arrillaga and Jones[11] found that yields of oil obtained in the distillation of lemongrass can be raised by closing the condenser valve of the still at frequent intervals during distillation. This allows pressure to build up with the result that the plant tissues are ruptured, liberating additional oil. Lemongrass oil distilled by the intermittent method proved to have a higher citral content than oil distilled in the usual way.

Coomber and Cosgrove[75] examined some samples of lemongrass oil from a distillery in Tanganyika in an effort to determine the cause of the low citral content of the oil produced there in 1944—1946. The samples contained 74—79% (v/v) of aldehydes whereas oil from this distillery normally contains 80% aldehydes as determined by the bisulfite method. Information supplied with the samples indicated that the grass was cut as often as every two months and immaturity of the grass was thought to be the main reason for the low citral content of the oil. Exhaustion of the soil on which the lemongrass was grown and the fact that in some instances the grass was partially dried before distillation may have been contributing factors. The oil distilled from grass which was cut soon after the end of the rainy season was particularly low in citral.

Naves[1] isolated a diterpene ketone from oil of lemongrass (*Cymbopogon flexuosus* Stapf.) produced in Guatemala. The ketone distilled at 157—158° at 1.7 mm., was optically inactive and had d^{20}_4 0.9464 and $n^{20}D$ 1.5206. It gave a 2,4-dinitrophenyl hydrazone m.p. 128° but did not readily yield other derivatives.

Lime Oil. Exports of lime oil from Mexico to the United States amounted to 77,000 lb. in 1947 or about the same quantity as in the preceding year. In 1948 exports of Mexican lime oil to the U.S.A. reached a total of 97,000 lb. and were valued at $376,000.

The amount of lime oil exported from the British West Indies in 1947 was the lowest in ten years. Table X shows exports of lime oil for 1946 and 1947.

Table X.

Exports of Distilled Lime Oil from the British West Indies[76]

	Quantity		Value	
	1946	1947	1946	1947
	Lb.	Lb.	£	£
Dominica	31,304	26,889	32,023	30,067
Grenada	8,680	7,687	10,802	10,131
Jamaica	21,635	12,275	30,752	11,995
Trinidad	16,604	1,925	22,343	6,291
Total	78,223	51,776	95,920	58,184

Kesterson and McDuff[63] analyzed six samples of coldpressed oil of Persian limes produced in Florida. The analytical values varied between the following limits: d^{20}_{20} 0.8798 to 0.8823, $n^{20}D$ 1.4842 to 1.4853, $\alpha^{20}D$ + 38.6° to + 41.8°, aldehyde content 3.66% to 5.52%, ester content 7.42% to 8.20% and evaporation residue 12.95% to 14.67%. Five samples of steam distilled lime oil, prepared by steam treatment of the peel after cold pressing, showed analytical values as follows: d^{20}_{20} 0.8556 to 0.8579, $n^{20}D$ 1.4743 to 1.4751, $\alpha^{20}D$ + 46.84° to + 50.52°, aldehyde content 1.61% to 2.71%, ester content 2.41% to 3.49% and evaporation residue 0.18% to 1.23%.

Linaloe Oil. ***See*** **Bois de Rose Oil.**

Lippia Asperifolia Oil. Naves[77] examined an essential oil distilled in Tanganyika from the flowering tops of the plant *Lippia asperifolia* Rich. The constants of the oil (average values for two samples) were as follows: d^{20}_{4} 0.922, $n^{20}D$ 1.5077 and $\alpha^{20}D$ — 6.1°. The head fraction of the oil contained a small amount of ocimene while the main fraction (roughly 80% of the oil) consisted of a mixture of aliphatic triene ketones analyzing for $C_{10}H_{14}O$. The ketone mixture on catalytic hydrogenation yielded 2,6-dimethyl-4-octanone, the product being identical with tetrahydrotagetone which was prepared by hydrogenating tagetone from the essential oil of *Tagetes glandulifera*. The ketonic fraction of *Lippia asperifolia* oil thus consists of dehydro-tagetones to which the name of "tagetenones" is given.

Melaleuca Alternifolia Oil. ***See*** **Tea Tree Oils.**

Melaleuca Bracteata Oil. Cosgrove *et al.*[78] examined a sample of essential oil prepared from the leaves and twigs of *Melaleuca bracteata* saplings which were grown in Kenya from seed obtained from New South Wales. The oil, obtained in a yield of 0.5%, had $d^{15.5}_{15.5}$ 1.0506, $\alpha^{20}D$ — 3.03°, $n^{20}D$ 1.5370 and was soluble in 1.5 volumes and more of 70% alcohol at 15.5°. The composition of the oil was found to be as follows:

Methyl eugenol	82.0%
Esters as cinnamate of $C_{10}H_{17}OH$	11.7%
Cinnamic aldehyde	1.0%
Eugenol	1.7%
Cinnamic acid	0.3%
Free alcohols as $C_{10}H_{17}OH$	2.0%
Terpenes and sesquiterpenes	1.3%

Another sample of *M. bracteata* oil from the same source which was examined by Mitchell[79] had a lower density (1.0430, 15.5/15.5) and contained 75% of methyl eugenol. Mitchell observed that the methyl eugenol fraction was odorless when first distilled and required several weeks to develop the characteristic fragrance. Both Cosgrove and Mitchell failed to detect the presence of cinnamyl alcohol in the oil and found evidence that the alcohol component is tertiary.

Melaleuca Viridiflora Oils. ***See*** **Niaouli Oil** ***and*** **Tea Tree Oils.**

Menthol, Brazilian. The curtailment of mint production in Brazil in the two seasons following the disastrous over-expansion of the menthol industry in 1944—45 brought about a gradual strengthening of the menthol market. As a result, farmers in the State of São Paulo increased their plantings of peppermint in the 1947—48 season (Table XI). The wholesale price of menthol in New York rose from $8 to $9 per lb. during the second half of 1947 and remained in the vicinity of $9 throughout 1948. Although this figure was well below the peak prices reached in 1944, it was much higher than the average price of menthol in the years before World War II.

Table XI.

Production of Peppermint Oil in the State of Sao Paulo, Brazil[80]

	Area Planted Alqueires *	Oil Produced Kg.
1942-43	6,756	13,300
1943-44	10,030	547,961
1944-45	38,020	1,033,474
1945-46	1,345	30,000
1946-47	1,690	40,000
1947-48	5,178	228,000

* 1 Alqueire = 5.98 acres.

Brazil exported 313 metric tons of menthol in 1947 as compared with 352 metric tons in 1946. The largest shipments of menthol during the first ten months of 1947 were destined for the United States (154.5 tons), the United Kingdom (80.5 tons) and France (14 tons). Stocks of crystalline menthol on hand in Brazil at the end of 1947 were estimated at 150 tons. Shipments of menthol from Brazil in 1948 amounted to only 135.6 metric tons, or less than half the total recorded in 1947.

Simmons[81] states that the peppermint oil which is exported from Brazil after partial removal of the menthol usually contains over 40% of free menthol and 10—15% of combined menthol. Normal samples of the oil show a specific gravity at 15° in the range 0.895 to 0.905, an optical rotation of — 26° to — 31° and a refractive index at 20° of 1.459 to 1.463. The oil is usually soluble in three to five parts 70% alcohol but occasional samples require from one to two volumes of 80% alcohol.

Menthol, Japanese and Chinese. Production of menthol in Japan in the three years after the war was far below the normal output. The area planted to mint in Hokkaido prefecture in 1946 amounted to only 185,100 hectares as compared with 1,298,000 ha. in 1940. Because of the continuing food shortage, farmers

were obliged to use much of the land formerly planted to mint for the production of food crops. This factor, together with the high cost of replanting the mint fields, prevented any large-scale attempts to rehabilitate the menthol industry.

The United States has been the principal market for Japanese menthol in the immediate post-war period. Imports of this commodity into the United States amounted to 14.3 tons in 1947 and 30.1 tons in 1948.

The 1947 harvest of peppermint in China gave an estimated yield of 300,000 lb. of oil, but the farmers were reluctant to sell the oil to the menthol factories because of the severe inflation. Only two menthol plants were operating in Shanghai in 1947 and their combined output of menthol for the year was 50,000 lb. During the first 11 months of 1947, exports of menthol from China totaled 61,676 kg. Shipments to the United States accounted for 38,916 kg. Production of menthol in Formosa in 1947 was reported to be insufficient to supply the needs of the island.

Micromeria Abyssinica Oil. Naves[82] examined a sample of the volatile oil distilled in Tanganyika from the flowering tops of the herb *Micromeria abyssinica* (Hochst.) Benth. The oil, which had d^{20}_4 0.9175, $n^{20}D$ 1.4781 and $\alpha^{20}D$ + 26.22°, was found to contain approximately 42% of *d*-isomenthone and 17–18% of *d*-pulegone. The *d*-isomenthone isolated from the oil had a specific rotation of + 93.85° and a refractive index of 1.4545 at 20°.

Mignonette Oil. The composition of the volatile portion of the concrete from mignonette flowers (*Reseda odorata*) was investigated by Sabetay *et al.*[83]. Codistillation with diethylene glycol yielded a yellow oil (acid value 56, ester value 95) containing crystals of paraffin hydrocarbons. The oil had a strong odor of coffee and mignonette which was due in part to esters as the odor was weakened by saponification. Eugenol, phenol, free caprylic acid, higher fatty acids and acetic acid esters were found to be present in the oil. An odorous fraction distilling at 135–155° at 11 mm. contained sulfur and nitrogen and gave an odor of styrene when heated with potassium hydroxide, indicating the possible presence of phenylethyl isothiocyanate. However, when the fraction was tested for this compound by treatment with alcoholic ammonia it failed to yield crystals of phenylethyl thiourea. (Cf. Schimmel Ber., Jubiläums–ausgabe 1929, 221.)

Mustard Oil. A bromometric method for the determination of mustard oil was proposed by Cavicchi[84]. The method is based on the reaction of allyl isothiocyanate with nascent bromine, a reaction which involves both bromination and oxidation and consumes 8 equivalents of bromine per molecule of allyl isothiocyanate. To carry out the determination the mustard oil solution is treated with an exactly measured excess of standardized potassium bromate solution and hydrochloric acid and potassium bromide are added. After 10–12 minutes the excess free bromine is determined by adding standardized potassium arsenite solution in excess and titrating back to the methyl orange endpoint with potassium bromate. This method gives slightly lower results than the Gadamer argentometric method which is official in the Italian and United States pharmacopoeias.

The acidimetric method for the determination of mustard oil which was proposed by Viebock and Brecher (Pharm. Monatsh. 11: 204, 1930) was modified by Böhme[85] who found that it gave results which were too low. The original method consisted in converting the sulfur of mustard oil (allyl isothiocyanate) to sulfate by means of alkaline hydrogen peroxide solution and titrating the excess alkali. In the modified method the allyl isothiocyanate is treated with ammonia to convert it to allyl thiourea which then reacts quantitatively with the alkaline peroxide solution. In analyses of mustard oil by this procedure Böhme secured results which were in close agreement with those obtained by the iodometric method of the Swiss Pharmacopoeia.

Myroxylon Pereirae Wood Oil. The wood of the tree from which Peru balsam is derived (*Myroxylon Pereirae* (Royle) Klotzsch.) yields an essential oil having *d*-nerolidol as the principal constituent, according to Naves[86]. The balsam, which is an exudate obtained by destroying a portion of the bark of the tree, contains around 2% of nerolidol, either free or esterified. Naves distilled the essential oil from two samples of *M. Pereirae* wood, one from an intact tree and the other from a tree which had been tapped for balsam. The bark, which contained no nerolidol, had been removed from both blocks of wood. The constants and constituents of the oils from the two trees, together with the yields, are shown below.

	Essential Oil from	
	Healthy Tree	Injured Tree
Yield	0.74%	1.08%
d^{20}_4	0.9096	0.8951
n^{20}_D	1.4928	1.4880
α_D	+ 7.21°	+ 7.92°
Cadinenes	28 — 30%	6 — 8%
d-Nerolidol (free)	50 — 52%	68 — 70%
l-Cadinol (free)	12 — 14%	7 — 9%
Alcohols (esterified)	2%	6%

It will be noted that the wood of the tapped tree gave a higher yield of oil than the wood of the healthy tree. Moreover, the oil from the injured tree contained the higher proportion of nerolidol. The cadinol present in *M. Pereirae* oil distilled at 125° at 2 mm. and when hydrogenated yielded the same dihydrocadinol (m.p. 132—133°) as did the cadinol isolated from cabreuva oil (see page 8). *M. Pereirae* wood oil differs in composition from cabreuva oil only in that it contains substantial amounts of cadinene but little or no bisabolol. The author suggests that trees which have been exhausted by being tapped for balsam be utilized for production of the wood oil as a source of nerolidol.

Niaouli Oil. Production of niaouli oil in New Caledonia in recent years has been on a very small scale because of a shortage of labor for picking the leaves of *Melaleuca viridiflora* from which the oil is distilled. Total exports of niaouli oil for 1946 amounted to only six metric tons but prices were more favorable in the following year and 11 metric tons of the oil were exported. In 1948 niaouli oil exports dropped again to seven tons.

Treatment of niaouli oil with lead oxide as specified by the French Codex of 1937 makes the oil more, rather than less, susceptible to deterioration, according to Guillot.[87] Storage tests showed the importance of filling containers of the oil to the top to exclude air. Diffuse light was not harmful to the oil in the absence of air but promoted oxidative changes when the oil was stored in partially filled bottles. A sample of untreated niaouli oil was unchanged after storage in a light room for nearly eight years in an uncolored glass-stoppered bottle filled nearly to the top.

Nim Blossom Oil. The essential oil of nim blossoms (*Melia azadirachta*) was isolated by Mitra *et al.*[88] by steam distillation of the alcohol or ether extract of the dried blossoms. The oil has a highly pungent odor but when diluted in alcohol develops a pleasing fragrance similar to that of the fresh flowers. The oil consists of sesquiterpene derivatives, the lowest boiling fraction having the composition $C_{15}H_{24}O_2$. Saponification of the oil gives a sweet smelling neutral component in a yield of 60% and an acidic fraction of pungent odor.

Ocimum Canum Oil. The possibility of growing camphor basil (*Ocimum canum* Sims) in Germany as a source of camphor was investigated during World War II at the Reichs-Institut für Forst- und Holzwirtschaft at Reinbek/Hamburg. A report on the work was made by Gerda Bruns-Runge[89]. The morphology of the *O. canum* plant was thoroughly studied at all stages of its development and the findings were in general agreement with earlier literature on the subject. Experimental cultivation of camphor basil for three seasons demonstrated that the plants would grow to maturity in the climate of northern Germany if started early in the spring. However, in order to get a satisfactory crop it was necessary to sow the seed in flats and then transplant the seedlings as the seed planted directly in the open fields did not come up well. The plants were analyzed for essential oil content by means of the Moritz microdistillation apparatus, the oil being distilled with xylene to prevent solidification of the camphor in the condenser. Determinations of the oil yield from separate parts of the blossoming plants showed that the highest concentration of oil occurs in the young, incompletely developed leaves. Yields of oil from the different parts of the plant (in vol.% based on the dry weight of the plant material) were as follows: full-grown leaves 5.94%, young leaves 8.12%, blossoms and buds 7.32% and fruits 3.53%. The roots and woody stems contained no oil. In an attempt to determine the best time to harvest camphor basil, the oil content of the whole plant exclusive of roots and woody stems was determined at intervals during the final stages of growth. The oil content (vol.%, dry basis) was 7.21% shortly before blossoming, 6.94% at the start of the blossoming period, 7.14% at full bloom and 5.48% after the seed had ripened. The oil content of the leaves alone was found to reach a definite maximum (5.37%) at the time when the plants were just starting to blossom.

Camphor basil was also grown successfully as an annual in Massachusetts by Youngken and Hassan[90]. The dried leaves and flowering tops harvested in October yielded an average of 2.51% of crystalline camphor and 2.5% of volatile oil having d^{25} 0.9225 and n^{25} 1.4735. The authors made a histological examination of the stem, leaf and floral parts of the plant which they designated as

Ocimum kilimandscharicum Gürke. (For a discussion of the confusion existing in regard to the botanical identity of camphor basil, see J. Am. Pharm. Assoc. 35: 153, 1945.)

Experiments to determine the influence of potassium on the accumulation of essential oil in the leaves of *Ocimum canum* were carried out by Kalinkevich[91] (cf. Schimmel Report 1946, 39). The plants were grown by the hydroponic method and were treated with solutions of varying K content before flowering and during the flowering period. When the N concentration was kept constant, the essential oil content of the leaves was found to vary inversely with the K content of the nutrient solution. In one experiment, plants were given the same treatment until ready to blossom and then divided into two groups, one being given increased K ($\frac{1}{2}$N—2K) and the other a reduced amount of K ($\frac{1}{2}$N—$\frac{1}{4}$K). After 20 days the essential oil content of the first group of plants was 2.8 vol.% and of the second group, 5.17 vol.%. Reducing sugars in the plants of the latter group amounted to 7.91% as compared with 3.58% in the plants receiving the higher amount of K. The introduction of dilute solutions of K salts into *O. canum* leaves by vacuum infiltration caused a decrease in essential oil content while infiltration of glucose and fructose tended to promote formation of essential oil.

Ocimum Gratissimum Oil. Dulou and Pétard[92] reported that a variety of *Ocimum gratissimum* L. which grows wild in the Society Islands and other islands of French Oceania contains an essential oil rich in eugenol. The plant, known as *Mivi Taratoni* or Caledonian basil, is a shrub which may grow to a height of 3 meters. Eight kg. of plant material (stems, leaves and blossoms) yielded 90 g. of an oil having a powerful odor, burning taste and physical constants as follows: d^{20} 1.0069, $[\alpha]^{20}_{D}$ — 6.23° and n^{20} 1.5293. The oil contained 61.8% of eugenol, 17.4% of other phenolic compounds and 20.8% of non-phenolic substances including ocimene and sesquiterpenes.

Ocotea Cymbarum (Brazilian Sassafras) Oil. United States imports of *Ocotea cymbarum* oil from Brazil in 1947 totaled only 383.5 tons or 217 tons less than in 1946. During the first eight months of 1948, receipts of *Ocotea cymbarum* oil amounted to 208.4 tons, so the total for the year was probably lower than in 1947. This oil was originally introduced to the American market to take the place of Japanese "artificial sassafras oil" as a source of safrole. When shipments of this product from Japan were resumed in 1947—48 the demand for the Brazilian oil became less acute and the price declined. From April to December of 1948 the wholesale price of *Ocotea cymbarum* oil on the New York market was approximately $0.55 per lb. as compared with $0.90 per lb. at the beginning of 1947.

Some of the minor constituents of *Ocotea cymbarum* oil were identified by Hickey[93]. The oil used in the investigation had characteristics as follows: d^{15} 1.076, n^{20}_{D} 1.5350, α^{20}_{D} — 1.42°, congealing point + 9.0°; soluble in 2.0 volumes of 90% alcohol at 20° and opalescent in 10.0 volumes. In addition to safrole (92.9%) the oil contained *l*-α-pinene (0.7%), eugenol (0.6%), *n*-valeraldehyde (0.001%), furfural (0.17%), cineol (0.21%), benzaldehyde (0.03%) and an unidentified com-

pound distilling at 215—225° at 758 mm. which had a camphoraceous odor. A hydrocarbon fraction which distilled at 110—114° at 1 mm. appeared on the basis of carbon-hydrogen determinations to consist of a sesquiterpene.

Shukis and Wachs[94] determined the congealing points of samples of *Ocotea cymbarum* oil containing various amounts of safrole and the data provide the basis for a cryoscopic method for the commercial evaluation of this oil. The samples were analyzed for safrole using the method of Huzita and Nakahara (J. Chem. Soc. Japan 62:5, 1911) in which safrole is determined as the mercuric chloride addition product. Rectified *Ocotea cymbarum* oil containing 91.9% of safrole congealed at 8.8° while pure safrole isolated from the oil congealed at 11.0° and the remaining oil (safrole content 69.1%) showed a congealing point of 2.4°. Mixtures of safrole with the oil gave congealing points intermediate between these values. The graph of congealing temperature vs. weight per cent safrole is a straight line and can be used to determine the safrole content of any sample of oil from the freezing point, providing this does not lie below 0°.

Opercularia Aspera. Lahey and Sutherland[95] demonstrated that the offensive odor of the shrub *Opercularia aspera* Goertn. is due to the presence of methyl mercaptan.

Orange Oil. Exports of orange oil from Italy in 1947 reached a total of 26.5 metric tons which was not far below the level of annual exports in 1937 and 1938. However, in 1948 Italian exports of orange oil decreased to 12.9 metric tons. The Italian product is meeting heavy competition in world markets from orange oil produced in Brazil, French Guinea, California and Florida. Comparative prices in New York in July 1948 were as follows: Italian orange oil $1.30 per lb., Brazilian $1.45, African $1.75 and California coldpressed, $1.40.

Shipments of sweet orange oil amounting to 159 metric tons were exported from French Guinea in 1947. Of this quantity France received 146 tons and the United Kingdom 13 tons. In 1948 total exports of orange oil from French Guinea amounted to 136 metric tons.

The Brazilian orange crop in 1947 was small as the result of the loss of trees from plant diseases. The output of orange oil amounted to only 40 to 50 tons as compared with 120 tons in 1946. Imports of Brazilian orange oil into the United States totaled 25 tons in 1947 and 18 tons in 1948.

Exports of orange oil from the island of Jamaica in the West Indies during the period 1941—1947 are shown below.

TABLE XII.

EXPORTS OF ORANGE OIL FROM JAMAICA[76]

	Lb.	£
1941	52,884	35,941
1945	31,457	14,202
1946	52,319	23,576
1947	55,975	30,961

Kesterson and McDuff[63] made a comprehensive study of the factors affecting the quality of the orange oil produced in different plants in Florida during the

1947—48 season. Four different types of equipment are used there for the production of coldpressed orange oil, a by-product of the citrus juice canneries. The Pipkin roll and the screw press are used to extract oil from the discarded peel while the Fraser Brace extractor removes the oil from the whole fruit by means of abrasive rolls. The Pipkin juice extractor squeezes the juice and oil from the orange in one operation. Each of these machines produces the oil in the form of an aqueous emulsion from which the oil is separated by centrifuging.

Thirty-five samples of orange oil were analyzed during the season, each representative of a week's production by one of four plants using the four different types of extractors. The oranges used for the production of oil from October to March were mixtures of the Hamlin, Pineapple and Parson Brown varieties and seedling oranges while those used during the remainder of the season were Valencias. The properties of the oil samples varied between the following extremes: d^{25}_{25} 0.8416 to 0.8458 (U.S.P. XIII limits 0.842 to 0.846), $n^{20}D$ 1.4718 to 1.4734 (U.S.P. 1.4723 to 1.4737), $\alpha^{25}D$ + 95.16° to + 97.76° (U.S.P. + 94° to + 99°), evaporation residue 1.07% to 4.93% (U.S.P. not less than 1.7%). The aldehyde content of the oils, determined by the hydroxylamine method, ranged from 0.92% to 2.04% (as decyl aldehyde) and the ester content from 0.04% to 1.63%.

Yields of oil in the different plants varied from 1.85 lb. per ton of peel to 9.70 lb. per ton, depending on the efficiency of the extraction process and the length of time the peel was left in the extractor. Some producers, in an effort to secure the maximum production of oil per day, extracted only a small proportion of the oil present in the peel and the product frequently failed to conform to U.S.P. specifications. Increasing the yield resulted in raising the specific gravity and refractive index of the oil and lowering the optical rotation. These changes show that more thorough treatment of the peel results in an increase in the proportion of constituents of high molecular weight and a corresponding drop in the percentage of optically active limonene in the oil. The data gathered during the 1947—48 season indicated that adjustment of the extraction process to yield 6.5 to 8.5 lb. of oil per ton of peel from midseason oranges would assure the production of orange oil of U.S.P. quality.

It was found that the use of an excessive amount of water during the extraction process resulted in a loss of aldehydes from the oil, to the detriment of its flavor value. Storage of the fruit for three to five days before extraction did not affect the physical constants of the oil but resulted in a slight decrease in the aldehyde content and an increase in the ester content and evaporation residue. The oil from Valencia oranges contained a higher proportion of aldehydes than the oil from mixtures of the other varieties. The aldehyde content of the Valencia orange oil began to decline after the oranges had passed the peak of maturity while the ester content continued to rise.

In the canning of orange juice a portion of the volatile oil from the peel is removed by heating the juice under vacuum in order to improve its flavor and keeping qualities. Analysis of nine samples of this distilled oil showed the characteristics to be as follows: d^{25}_{25} 0.8400 to 0.8464, $n^{20}D$ 1.4715 to 1.4732, $\alpha^{25}D$

+ 95.92° to + 98.56°, aldehyde content 1.72% to 2.18%, ester content 0.22% to 1.38% and evaporation residue, 0.08% to 1.21%. It will be noted that the distilled oil is richer in aldehydes than the coldpressed oil, the explanation being that aldehydes originally present in the juice are removed along with the peel oil during vacumm distillation.

Three additional aldehydes of high molecular weight were found by Naves[96] in sweet orange oil from French Guinea. In the fractions distilling between 80° and 102° at 2.5 mm., 2-decenal and dodecanal (lauric aldehyde) were identified as the semicarbazones, m.p. 162° and 105°, respectively. Oxidation of the aldehydes in a fraction distilling in the range 90—130° at 10 mm. by means of silver oxide yielded a mixture of acids, one fraction of which gave decanoic acid on ozonolysis and dihydroxylauric acid on treatment with alkaline potassium permanganate. These reactions furnished proof of the presence of 2-dodecenal in the oil. 2-Decenal and 2-dodecenal are apparently destroyed by treatment of orange oil with sodium bisulfite.

The constants of the sweet orange oil produced in Palestine are given by Nicholls[97] as follows: $d^{15.5}_{15.5}$ 0.850 to 0.852, α_D + 97° to + 99°, n^{20} 1.473 to 1.4745; residue at 100°, 3.2% to 3.8%. The oil contains between 1.5% and 1.9% of aldehydes calculated as decanal, approximately 90% of *d*-limonene, traces of *d*- and *l*-terpineol, linalool, nonyl alcohol and caprylic esters. As the proportion of oxygenated constituents in Palestinian orange oil is fairly high (4.9—5.4% soluble in diacetin) it is economically feasible to prepare the sesquiterpeneless oil. The yield is usually about 2.5% of the original oil.

Thirteen samples of sweet orange oil from Palestine were analyzed by Islip and Major[98] at the British Imperial Institute. In most cases the samples showed a specific gravity within the range given by Nicholls (see above), the extreme values being 0.8490 and 0.8565 (15.5°/15.5°). The samples conformed closely to his description in respect to refractive index and in most cases in respect to optical rotation, although a few samples showed α^{20}_D below + 97°. The lowest rotation observed was 94.47°. Of the 13 samples, eight contained more than 3.8% of non-volatile matter and in two cases the amount was over 6%. The aldehyde content of the samples examined at the Imperial Institute ranged from 0.94% to 1.37%, whereas Nicholls gives 1.5% as the minimum aldehyde content of Palestinian orange oil. However, his method of assay for aldehydes involves more drastic conditions for the reaction with hydroxylamine hydrochloride and may give rather high values.

Origanum Virens Oil. Costa and Cardoso do Vale[99] studied the chemical composition of the essential oil of *Origanum virens* Hoffms. and Link of Portuguese origin (cf. Schimmel Report 1916, p. 46). Pinene, cineol, camphor, thymol and carvacrol were identified in the oil and the presence of dipentene was suspected. Acetylation indicated that the oil contained a substantial amount of alcohols but these were not identified.

Pæsia Scaberula Oil. Briggs and Sutherland[100] reported that the scented fern *Pæsia scaberula* which is native to New Zealand contains terpene derivatives, a rare occurrence in the order of ferns. Steam distillation of fronds collected in the autumn gave a 0.05% yield of an essential oil which was deep red in color and had d^{25}_4 0.948 and $n^{25}D$ 1.4990. From the oil were isolated the hydrocarbon *n*-heptacosane and a dicyclic sesquiterpene alcohol, $C_{15}H_{24}O$, having d^{25}_4 0.982 and $n^{25}D$ 1.517. The presence of oxygenated terpenes in the oil was also indicated.

Palmarosa Oil. Exports of palmarosa oil from India in the year ending March 31, 1948, amounted to 2,708 gal. and were valued at 393,385 rupees. In the following year exports of the oil increased to 7,950 gal. valued at 1,005,473 rupees.

Palmarosa (motia) oil produced in the Elichpur section of Berar Province in India has a more sweet and roselike odor than the palmarosa oil from the Hyderabad region, according to Narian and Das Gupta[101] who analyzed several samples of oil from each locality. The Elichpur samples contained 78% to 87% of free alcohols as geraniol and 4% to 7% of esters as geranyl acetate while the Hyderabad samples contained 62% to 68% of free geraniol and 11% to 15% of geranyl acetate. The solubility in 60% alcohol was shown to be a good index of the geraniol content of palmarosa oil. The high-geraniol oil from Elichpur was soluble in from 2.1 to 4.2 volumes of 60% alcohol while the Hyderabad oil was insoluble in up to 10 volumes of 60% alcohol.

The motia and sofia varieties of *Cymbopogon Martini* Stapf which yield palmarosa and gingergrass oils, respectively, were cultivated experimentally by Subba Rao *et al.*[102] in South India. Equal quantities of seeds of the two varieties were sown but the motia seed germinated poorly in comparison with the sofia seed. After 1½ months the young plants were set out about 6 in. apart and fertilizer was applied shortly before they were ready to blossom. The grass was cut a week or ten days after flowering started, 288 lb. of grass being obtained from 682 motia plants and 1082 lb. of grass from 1070 sofia plants. One lot of sofia plants which was not fertilized gave a much lower yield of grass than the fertilized plants. The yield of palmarosa oil from the fresh motia grass was 0.16% (yield per acre 47.3 lb.) and the yield of gingergrass oil from the fresh sofia grass was 0.176% (yield per acre 113.7 lb.). The total geraniol content of the motia oil was 87% and of the sofia oil, 56%. The flowering tops of motia grass gave 0.52% of oil, the leaves 0.20% and the stalks 0.012%. The oil obtained from the flower buds of both varieties of *C. Martini* has a finer odor than oil from the other parts of the plants.

Patchouli Oil. In the years 1946–48, imports of patchouli oil into Malaya exceeded exports. In 1947 exports of the oil totaled 32,059 lb. and imports 46,812 lb. Trade in patchouli oil was more active in 1948, with exports amounting to 48,481 lb. and imports to 55,901 lb. Almost all of the patchouli oil imported into Malaya is produced in Sumatra while the remainder originates in Java. Patchouli oil was selling in the United States for $35 per lb. or even more in the early months of 1947 but the price dropped rapidly and by the end of 1948 was in the vicinity of $5 per lb.

Exports of patchouli oil from the Seychelles decreased from 9,005 kg. valued at Rs. 630,715 in 1947 to 1,927 kg. valued at Rs. 91,115 in 1948.

Pectis Papposa Oil. Bradley[103] reported that *Pectis papposa*, a desert plant which grows in southwestern United States from New Mexico to California, contains an essential oil rich in cuminaldehyde. *Pectis papposa* is a bushy plant, about a foot high, which has bright yellow blossoms and dark green foliage. Oil glands are visible along the margins of the leaves. The seeds germinate following a rain or irrigation of the land and the plants reach maturity quickly. *Pectis papposa* plants gathered in June yielded 0.60% of essential oil while plants gathered late in December yielded only 0.40% of oil. Constants of the oil were as follows: d^{25}_4 0.939, $n^{25}D$ 1.4992 and $\alpha^{25}D$ + 16.3°. Analysis of the oil showed that it contained approximately 50% cuminaldehyde, 20% pinocarvone, 5% carvone and 25% of a bicyclic terpene which was probably β-pinene.

Pepper Oil. Gokhale *et al.*[104] isolated a fragrant essential oil from the fruits of long pepper (*Piper longum*) by steam distillation of the ether extract. The oil distilled in the range 260—280° and had d^{28} 0.8210, n^{28} 1.417, $[\alpha]D$ — 22° and an acetyl value of 146. The oil gave negative tests for phenols, aldehydes, ketones, esters, free acids and nitrogenous compounds.

Pepper Tree Oil. The leaves and twigs of the Peruvian pepper tree (*Schinus molle* L.) were distilled with steam in a California laboratory, yielding 0.50% of essential oil. Kidder[57] gives the constants of the oil as follows: d^{25}_{25} 0.849, α^{20} + 58°28′, n^{20} 1.4747, ester value 48.7 and ester value after acetylation 113.4. The leaves and twigs exude an oleoresin of pleasing odor which is most abundant during the fall months when the tree is producing pollen.

Peppermint Oil, American. Peppermint plantings in some areas of Indiana and Michigan did not survive the winter of 1947—48 and the crop acreage was considerably reduced in these states (see Table XIII). The total acreage in Oregon in 1948 was higher than in the previous year, despite the destruction of some mint fields in Columbia County by spring floods. The 1947 crop of peppermint yielded 1,603,000 lb. of oil and the 1948 crop yielded 1,551,000 lb. The average price per pound received by growers dropped from $6.94 in 1947 to $6.27 in 1948.

Table XIII.

Production of Peppermint Oil in the United States[105]

State	Acreage		Oil Produced		Value	
	1947	1948	1947	1948	1947	1948
	Acres	Acres	Lb.	Lb.	$	$
Indiana	18,500	14,300	574,000	500,000	1,018,000	3,425,000
Michigan	13,500	10,000	270,000	220,000	1,850,000	1,507,000
Oregon	11,000	12,600	550,000	567,000	3,850,000	3,232,000
Washington	4,400	4,800	209,000	264,000	1,411,000	1,544,000
Total	47,400	41,700	1,603,000	1,551,000	11,129,000	9,708,000

The major portion of the peppermint oil produced in the United States is consumed domestically. Exports of the oil dropped from 223.3 tons in 1947 to 114.7 tons in 1948.

Table XIV.

Exports of Peppermint Oil from the United States[106]

Destination	1947 Lb.	1948 Lb.
Canada	35,324	55,873
Mexico	26,286	16,000
Cuba	21,826	7,087
Brazil	23,690	18,438
United Kingdom	176,123	28,500
Netherlands	27,277	40,178
France	26,059	18,488
Australia	45,244	2,344
Other countries	64,834	42,439
Total	446,663	229,347
Value	$3,534,446	$1,735,183

The results of analyses of many samples of peppermint oil produced in the states of Oregon and Washington over a period of four years were reported by Tornow and Fischer[107]. The oils produced in the four different mint-growing districts, designated by the authors as oil types C, E, S and K, are described as being distinct from each other in odor and flavor but the analyses show no striking variations in menthol and ester content among these oils. Peppermint oil of type C is produced on both the Oregon and Washington sides of the Columbia River not far from the coast where the mint is grown on swampy land drained by ditches. Type E is produced along the upper Willamette and lower McKenzie rivers in Oregon and type S in the central section of the Willamette Valley. Oil of type K is distilled from mint grown on irrigated land in eastern Washington where the climate is dry. The average analytical values found for each type of oil (before rectification) are given in Table XV.

Table XV.

Average Constants of Oregon and Washington Peppermint Oils (Unrectified)

Type of Oil	Number of Samples	Esters %	Total Menthol %	$\alpha^{25}D$	d^{25}_{25}	n^{20}
C	337	5.60	53.90	−23.5°	0.9018	1.4629
E	135	7.57	59.21	−24.7°	0.9020	1.4621
S	68	7.36	57.05	−23.03°	0.9032	1.4633
K	519	6.34	53.06	−16.7°	0.9086	1.4651

The specifications of the U.S. Pharmacopoeia XIII for peppermint oil are as follows:

Ester content	not less than 5%
Total menthol content	not less than 50%
$\alpha^{25}D$	−18° to −32°
d^{25}_{25}	0.896 to 0.908
n^{20}	1.4590 to 1.4650

The average analytical values for the C, E and S types of oil (Table XV) met these requirements, although the values for individual oils in some cases fell outside the U.S.P. limits. The U.S.P. requirement that peppermint oil should be clearly soluble in 3 volumes of 70% alcohol was met by over 95% of the samples of these three types of oil.

ARTEMISIA ABSINTHIUM (WORMWOOD).

OCIMUM BASILICUM (SWEET BASIL).

While the oils of type K on an average basis conformed to the standards for ester and total menthol content, they showed average physical constants deviating somewhat from the U.S.P. range. Preliminary experiments indicated that rectification of the oil would correct the specific gravity and refractive index but would not bring the optical rotation within the specified range. Fifteen per cent of the samples of type K oil failed to pass the test for solubility in 70% alcohol.

The relationship between the maturity of peppermint plants at the time of harvesting and the yield and quality of the oil distilled from the plants was studied by Bullis *et al.*[108] at the Oregon Agricultural Experiment Station. It was found, in experimental distillations carried out during the 1944 and 1945 seasons, that the yields per acre of dry hay and oil increased as the plants matured until they were nearly in full bloom, after which the dry hay yield remained nearly constant while the oil yield decreased slowly. The oil content of the hay, calculated on a moisture-free basis, decreased slowly from the first to the last sampling. The number of oil glands on the leaves increased steadily until the plants were in full bloom, about 80% of the glands being located on the lower surfaces of the leaves.

The peppermint oil gradually grew richer in total menthol and in esters from early in the season until a month after the plants had reached full bloom. The oil distilled from plants harvested on August 1, 1945 had a total menthol content of 45.6% and an ester content of 3.75% while the oil from plants harvested on September 26 contained 63% of menthol and 8.46% of esters. The specific rotation of the peppermint oil distilled in midseason was lower than that of the samples distilled early or late in the season, but no consistent variations in specific gravity or refractive index of the oil according to the time of harvest were observed. Weathering of the mint hay in the field for a week after cutting did not materially affect either the yield or quality of the oil when the shattered leaves were saved and distilled with the sample.

Peppermint Oil, Palestinian. Halasz and Katz[109] reported that peppermint of the Mitcham type is grown in several places along the coastal plain of Palestine. The oil, obtained in a yield of 0.38%, has a density of 0.900 to 0.920, a specific rotation of — 16.4° to — 20.4°, and is soluble in 4.15 volumes of 70% alcohol. It has a total menthol content of 41% to 45% and an ester content of 5.4% to 15% as acetates.

Peppermint Oils. ***See also*** **Menthol.** Schmidt[110] attempted to determine the identity of the substances which give to the peppermint oil derived from *Mentha piperita L.* an aroma far superior to that of peppermint oil from *M. arvensis* Holmes (Japanese peppermint). These substances are concentrated in the tail fraction from steam rectification of the oil, this fraction having approximately the same chemical and physical properties whether obtained from Russian, Italian, Bulgarian or American peppermint oil. The following new constituents were isolated from the high-boiling fraction: jasmone $C_{11}H_{16}O$, an odorous sesquiterpene ketone $C_{15}H_{24}O$, a sesquiterpene alcohol $C_{15}H_{26}O$, *l*-caryophyllene (also present

in Japanese peppermint oil) and a bicyclic sesquiterpene showing a positive optical rotation. Menthofuran, a dextrorotatory terpene oxide previously found in French and Italian peppermint oils, was isolated in larger amounts from the American oil, the aroma of which is due in part to this constituent. Menthofuran distills with the menthol and menthone fractions but its autoxidation product, m.p. 186°, was isolated in small amounts from the tail fraction.

Menthofuran was also isolated from American peppermint oil by Bedoukian[111]. The maleic anhydride addition product had a melting point of 133° which was not depressed by admixture with the maleic addition product of synthetic menthofuran.

Garnero, Bénezet and Igolen[112] investigated the nature of the alcohols present in Brazilian peppermint oil which is derived from *Mentha arvensis* D.C. var. *piperascens* Malinvaud. A large quantity of dementholized peppermint oil (d^{15}_{15} 0.8938, $n^{20}D$ 1.4588 and αD — 32.5°) which still contained 44.7% of menthol was fractionated and the alcohols were isolated from the head fractions in the form of their boric acid esters. The following alcohols were identified: methanol, ethanol, *n*-butanol, 2-methyl-1-butanol, 3-methyl-1-butanol, 3-hexene-1-ol and 3-octanol. A mixture of saturated C_6 alcohols b.p. 150—151° at 732 mm. appeared to consist of *n*-hexanol and 3-methyl-1-pentanol. The amount of 3-octanol isolated was equivalent to 1.1% of the peppermint oil.

A color test developed by Fernandez Cabrera[113] allegedly serves to distinguish between peppermint oils of different geographical origin. The test procedure is as follows. Dissolve a few crystals of chloral hydrate in 1 cc. ethyl alcohol, add 0.5 to 1 cc. of peppermint oil and 1 cc. of 10% sulfuric acid and heat the mixture on the water bath at 60° for 10 minutes. Add a drop of concentrated nitric acid, heat the mixture again on the water bath for 15 minutes and then observe the color of the oily layer. Italian peppermint oil gives a green color while the American oil gives yellow and the German oil, yellow brown.

In studies on the nutritional requirements of *Mentha piperita*, Birkeli[114] found that black peppermint requires large quantities of potassium and nitrogen and a relatively smaller supply of phosphorus. The margin between maximum nutritive effect and toxicity seemed to be much narrower with potassium than with phosphorus and nitrogen. A positive correlation was observed between increasing supply of magnesium and the volatile oil content of the plants while a negative correlation was found between increasing supply of nitrogen, phosphorus or potassium and the oil content.

Petitgrain Oil. Paraguay exported 154.2 metric tons of petitgrain oil in 1947 or about one-half the quantity exported in the preceding year. In 1948 exports of petitgrain oil were somewhat higher, the total being 188.4 tons. The United States was the destination of a large proportion of the oil. Imports of Paraguayan petitgrain oil into the U.S.A. totaled 119 metric tons in 1947 and 152 tons in 1948. The declared value of these imports for the two-year period was $1,493,000.

BIOLOGICAL

In October 1917 the Bank of Paraguay fixed the minimum price for petitgrain oil at $6 per kilogram, f.o.b. Buenos Aires, and this minimum remained in effect during most of 1918.

Phyllocladus Trichomanoides Oil. The essential oil from the leaves of *Phyllocladus trichomanoides*, a New Zealand tree, was analyzed by Briggs and Sutherland[115]. Leaves collected in summer gave 0.13% oil having d^{25} 0.8876, $n^{25}D$ 1.4839 and $[\alpha]^{20}D$ + 8.48°. The following substances were isolated from the oil: *d*- and *dl*-α-pinene, myrcene, γ-terpinene, copaene, cadinene, phyllocladene, isophyllocladene, two unidentified diterpenes of which one was a solid and the other a liquid, and a dicyclic sesquiterpene alcohol having b.p. 134–137° at 5 mm., $n^{25}D$ 1.5040, d^{25} 0.9674 and $[\alpha]D$ – 9.5°. This alcohol apparently was a new cadinol as it gave *l*-cadinene dihydrochloride on treatment with hydrogen chloride in acetic acid. The oil from leaves of *P. trichomanoides* collected in winter contained α-pinene, dipentene, phyllocladene, isophyllocladene, a dicyclic sesquiterpene b.p. 122–127° at 10 mm. which may have been either calamene or impure cadinene, and an unidentified tricyclic sesquiterpene. This hydrocarbon, possibly a new compound, had b.p. 110–111° at 10 mm., d^{25} 0.9189, $n^{25}D$ 1.4928 and $[\alpha]D$ + 20.0°. It gave a crystalline nitrosite melting at 92.5–94° and yielded cadalene on dehydrogenation.

Pine Needle Oils. ***See also*** **Araucaria Excelsa Oil** ***and*** **Dacrydium Kirkii Oil.** the accumulation of large quantities of pine trimmings and Bailey[21] investigated the possibility of using this material for the production of pine needle oil. The main species of pine in the valley are shortleaf pine (*Pinus echinata*), Virginia pine (*P. virginiana*) and loblolly pine (*P. taeda*). In trial distillations of the fresh foliage from trees which were dominant in natural stands, Virginia pine and shortleaf pine foliage yielded approximately 0.3% of oil in October and November and 0.2% in February and March. Loblolly pine gave a yield of 0.35% of oil in January. The foliage from young Virginia and shortleaf pines grown in plantations gave average oil yields of 0.09% in June, 0.21% in October and November and 0.15% in February and March. Yields from the young trees were thus appreciably lower than yields from older (dominant) trees in natural stands. Composite samples of the needle oils from the three varieties of pine showed characteristics as follows: Virginia pine d^{15} 0.877, n^{20} 1.1797, α – 10.2°; shortleaf pine d^{15} 0.883, n^{20} 1.4832, α – 12.5°; loblolly pine d^{15} 0.871 and n^{20} 1.1712. The production of pine needle oil on a profitable basis under the conditions existing in the Tennessee Valley would depend on the use of mechanized harvesting equipment and a portable distillation unit to eliminate the cost of transporting the branches.

Pine Needle Oils. ***See also*** **Araucaria Excelsa Oil** ***and*** **Dacrydium Kirkii Oil.**

Pinus Maritima Resin Oil. Caryophyllene and longifolene were shown by Dupont *et al.*[116] to be the main components of the sesquiterpene fraction of turpentine oil from the maritime pine. Caryophyllene was identified by the formation of a blue nitrosite (m.p. 111–115°) and a nitrosate (m.p. 159°) and by conversion to caryophyllene alcohol (m.p. 95–96°) on treatment with acetic-sulfuric acid mixture. Longifolene was identified by means of the hydrochloride (m.p.

59–60°) and the formation of longifolic acid (m.p. 152–153°) on chromic acid oxidation.

Pinus Pumilio Oil. The annual production of *Pinus pumilio* oil in the Austrian Tyrol was reported to be around 6,000 kg. in 1947 and 1948. Thus it was only approximately half the average annual production before the war. A lack of demand for the oil brought about a reduction in price and in December 1948 the oil was quoted at $2.10 per lb. at the source.

Pinus Silvestris Wood Oil. A tricyclic diterpene ketone, $C_{20}H_{30}O$ (+ H_2 ?), was isolated by Sörensen and Bruun[117] from the wood of *Pinus silvestris* L. It was separated as the semicarbazone (m.p. 223–224°) from the neutral fraction of the oil obtained by distillation of the wood with superheated steam. The ketone (m.p. 50–52°) was given the name "cryptopinone".

Pinus Torreyana Resin Oil. The composition of the gum turpentine of Torrey pine (*Pinus torreyana* Parry) was determined by Haagen-Smit *et al.*[118]. The pine oleoresin yielded 17% of volatile oil having $d^{23.5}_4$ 0.8360, $n^{25}D$ 1.4656 and $[\alpha]^{25}D$ − 118°. The oil was found to consist of 75% *l*-limonene, 4% longifolene, 10% *n*-decyl aldehyde, 0.2% lauryl aldehyde, 5% *n*-undecane and 0.2% of an unidentified C_{10} carbonyl compound. Traces of nonane and *n*-heptane were also detected. The presence of paraffin hydrocarbons in this oil is not surprising since paraffins also occur in the turpentines of *Pinus coulteri* and *Pinus sabiniana*, two pines belonging to the same morphological group (*Macrocarpae*) as *Pinus torreyana*.

Prangos Ferulacea Oil. Khrimlian[119] determined the yield of essential oil from various parts of the plant *Prangos ferulacea* (L) Lindl. The seeds, or fruits, gave the highest yield (0.5%) while the blossoms gave 0.135%, the stems 0.015% and the leaves, 0.186%.

Radish Seed Oil. A new mustard oil containing the sulfoxide radical was isolated from radish seed (*Raphanus sativus* L., var. *alba*) by Schmid and Karrer[120]. The oil, which was named "sulforaphen", occurs in the form of a water-soluble glucoside. Boiling the ground radish seed in dilute acetone and evaporation of the acetone gave an aqueous solution of the glucoside from which the mustard oil was liberated by adding silver nitrate and treating the silver salt with sodium thiosulfate (Gadamer, Arch. Pharm. 237: 507, 1899). After purification by high vacuum distillation and chromatography, sulforaphen was obtained as a colorless oil distilling at 125–130° at 0.015 mm., $[\alpha]D$ − 108° in chloroform, − 136° in alcohol. Analysis showed that it had the molecular formula $C_6H_9ONS_2$, and the reactions of the compound led to the conclusion that it was 1-methylsulfinyl-3-butenyl isothiocyanate, $CH_3\overset{O}{S}CH{:}CHCH_2CH_2NCS$. On treatment with alcoholic ammonia, sulforaphen gave the corresponding thioureide, m.p. 219–220° (decomp.). On oxidation with fuming nitric acid it yielded methylsulfonic acid and when treated with silver oxide formed N,N'-di-[1-(methylsulfinyl)-3-butenyl] urea, m.p. 112–113°. The optical activity of sulforaphen was shown to be due entirely to asymmetry around the sulfoxide group.

In the purification of sulforaphen, a small amount of the corresponding nitrile, $CH_3S\overset{O}{\overset{\|}{C}}H{:}CHCH_2CH_2CN$, was isolated as an oil distilling at 120–125° at 0.001 mm., $[\alpha]^{13}D$ — 196° in alcohol. Other plant materials beside radish seed are known to produce both a mustard oil and the corresponding nitrile. For example, *Nasturtium officinale* yields phenylethyl isothiocyanate and phenylethyl cyanide.

Sulforaphen exhibited weak antibiotic activity when tested against several microorganisms and completely inhibited the germination of cress seed at a dilution of 1:1000. The nitrile showed no antibiotic properties.

Rose Leaf Concrete. Maranca[121] extracted a concrete from the leaves of the May rose (thorny variety) using trichlorethylene as the solvent. The concrete was a hard wax of a dark olive-green color. The absolute, prepared by washing the concrete with alcohol and evaporating the alcohol, was a very thick green liquid having the characteristic odor of rose leaves. The portion of the concrete which was volatile with ethylene glycol had a finer odor than the absolute.

Rose Oil. Spring frosts and drought injured the rose crop in Bulgaria in 1947 and weather conditions were also unsatisfactory in 1948. In both years the output of rose oil was less than 800 kg. whereas annual production before the war ranged from 2500 to 3500 kg.

United States imports of rose oil from Bulgaria amounted to 20,570 oz., valued at $560,900, in 1947 but dropped to only 2,420 oz. in 1948.

The 1948 rose harvest in the Grasse region of France yielded 500,000 kg. of rose blossoms, a small crop in comparison with the annual harvest before the war This quantity of blossoms should yield approximately 600 kg. of rose absolute.

Exports of rose absolute from France totaled 675 kg. (Frs. 37,246,000) in 1947 and 955 kg. (Frs. 48,167,000) in 1948.

Rosemary Oil. A sample of rosemary oil distilled from plants collected near Seulo in Sardinia was examined by Rattu and Tuveri[122]. The oil had d^{15} 0.9118, $n^{23.6}D$ 1.471, $[\alpha]^{22}D$ + 21°54′ and was soluble in 20 parts of 80% alcohol. The composition of the oil was as follows: free borneol 18.8%, bornyl acetate 17.2%, camphor 9.8%, cineol 9.2% by volume, and terpene hydrocarbons, including pinene, approximately 44% (by difference). Another sample of Sardinian rosemary oil examined by Puxeddu in 1925 (Ann. chim. applicata 15: 159 and 170) contained only 10.3% of free borneol and 4.6% of bornyl acetate.

Salvia Lavandaefolia Oil. Spanish sage oil, which is derived from *Salvia lavandaefolia*, contains camphor and cineol in proportions varying with the geological age of the soil in which the plant grows, according to Fernández and Abeniacar[123]. Tertiary eocene soils favor the production of camphor and quaternary alluvial soils favor the production of cineol. Samples of sage oil from three regions of Spain were found to contain neither borneol nor thujone.

Salvia Leucophylla Oil. Youngken and Heaps[124] isolated the aromatic constituents of *Salvia leucophylla* Greene, a variety of sage which grows wild in

California. Steam distillation of the air dried leaves yielded 2% of solid camphor and 2.9% of a volatile oil having n^{26} 1.4650 and d 0.9102.

Sandalwood Oil, Australian. Exports of sandalwood oil from Australia in the year ending June 30, 1947, totaled 28,723 lb. and had a value of £Aus59,145. Exports to the United Kingdom accounted for 12,320 lb.

Sandalwood Oil, East Indian. The Sandalwood Oil Factory of the Government of Mysore, India, produced approximately 120,000 lb. of sandalwood oil in 1947. There are six other small plants in India which have a combined annual output of 40,000 to 50,000 lb. of sandalwood oil. Exports of the oil from India during the year ending March 31, 1948 amounted to 71,899 lb., a slight decrease from the preceding year. In 1948-49, exports of the oil dropped to 31,019 lb.

Table XVI.

Exports of Sandalwood Oil from India[125]

Destination	Quantity		Value	
	1947-8	1948-9	1947-8	1948-9
	Lb.	Lb.	Rupees	Rupees
United Kingdom	40,725	9,750	1,430,894	325,000
Anglo-Egyptian Sudan	9,749	3,694	456,561	135,412
United States	7,450	3,540	298,055	136,960
Other countries	13,975	14,035	617,581	499,857
Total	71,899	31,019	2,803,094	1,097,229

Sassafras Oil, Brazilian. *See* **Ocotea Cymbarum Oil.**

Spearmint Oil. An exceptionally large crop of spearmint was produced in the United States in 1948. Growers in Indiana increased their spearmint plantings from 9,800 acres in 1947 to 13,000 acres in 1948 and Michigan farmers added 2,200 acres of new spearmint plantings to the 4,500 acres cultivated in 1947. In some cases the spearmint was planted in fields where peppermint was dying out so a part of the increase in spearmint acreage took place at the expense of peppermint acreage.

Total production of spearmint oil in 1948 amounted to 710,000 lb., an increase of 229,000 lb. over the preceding year. Production in Indiana accounted for 442,000 lb. of the 1948 total, the remainder being produced in Michigan. The abundant supply of spearmint oil brought a decline in the market value and producers received an average price of only $4.65 per lb. as compared with $5.60 per lb. for the oil from the 1947 crop.

Exports of spearmint oil from the United States rose from 30 tons valued at $436,600 in 1947 to 39 tons valued at $510,800 in 1948. Canada provides the principal export market for spearmint oil.

Spruce Needle Oil. Spruce needle oils of Rumania were prepared and characterized by Matei and Cocea[126]. Branches of *Abies alba* Mil. and *Picea excelsa* Lk. (Norway spruce) cut from trees of different ages were subjected to steam distillation for 14—16 hours. The highest yield of oil (0.97%) was obtained from adult spruce trees and the lowest (0.2%) from young Norway spruce trees. The

oil from young and adult spruce trees had d^{20} 0.8870, $n^{20}D$ 1.4746, $[\alpha]D$ − 42.9° and contained 15.8% of bornyl acetate. The oil from old spruce trees had slightly different constants but contained about the same amount of bornyl acetate. The fraction of spruce oil distilling in the range 150–170° and constituting 58% of the oil contained pinene (4%), camphene (15%) and carene (5%). Adult Norway spruce trees yielded an oil having d^{20} 0.8958, $n^{20}D$ 1.4743, $[\alpha]D$ − 39.54° and a bornyl acetate content of 20.8%. The oil from young Norway spruce contained 17.2% of bornyl acetate.

Summer Savory Oil. The characteristics of the oil distilled in California from a crop of summer savory which had been damaged by frost were given by Kidder[57]. The oil, obtained in a yield of 0.50%, had d 0.8807, α − 0°20′, n 1.4887, acid value 1.43, saponification value 25.2 and was soluble in 11 parts of 80% alcohol. The phenol (carvacrol) content of the oil was 25%. The low specific gravity indicated that the oil had a higher terpene content than summer savory oil of European origin but this might be reduced by discarding the head fractions of the oil.

Sweet Flag Oil. *See* **Calamus Oil.**

Tangerine Oil. Two samples of tangerine oil produced in Florida, one cold-pressed and the other distilled, were analyzed by Kesterson and McDuff[63]. The coldpressed oil had d^{25}_{25} 0.8456, $n^{20}D$ 1.4734, $\alpha^{25}D$ + 91.18°, aldehyde content 1.08%, ester content 0.34% and evaporation residue 1.53%. The distilled oil showed d^{25}_{25} 0.8407, $n^{20}D$ 1.4720, $\alpha^{25}D$ + 93.67°, aldehyde content 1.24%, ester content 0.25% and evaporation residue 0.20%.

Tea Tree Oils. Seasonal variations in the oil yield from individual tea trees of the species *Melaleuca alternifolia* Cheel were studied by Penfold *et al.*[127]. The oil yield was found to be higher in summer than in winter, a sudden increase in yield appearing in November, the first month of summer in New South Wales. The cineol content of the winter oil was slightly higher than that of the summer oil.

The existence of physiological forms of *M. alternifolia* which produce oils of much higher cineol content than the type species was disclosed by the same investigators[128]. In a study of 49 specimens growing in various localities it was found that only 19 trees yielded typical oils containing 6–14% of cineol. Fourteen trees gave oils containing between 30% and 45% of cineol while the remainder gave oils having a cineol content of 54–64%. The "medium cineol" form of *M. alternifolia* was designated as variety A and the "high cineol" form as variety B. The range of optical rotation for the oils of low cineol content was + 6.45° to + 8.58°; for the oils of medium cineol content, + 3.40° to + 5.50°; and for the oils of high cineol content, + 1.64° to + 2.90°. Thus the optical rotation can be used to estimate the amount of cineol present in oils of *M. alternifolia*.

The tree *Melaleuca viridiflora* Sol., which is found along the eastern coast of Australia, yields an oil rich in nerolidol and linalool. Penfold *et al.*[129] grew a number of plants of *M. viridiflora* from seed and harvested the foliage when the trees were three years old. The oil, obtained in yields of 1.7% to 2.6%, had d^{15}_{15} 0.8806 to 0.8857, n^{20} 1.4720 to 1.4763 and α + 13.3° to + 14.6°. It contained

approximately 30% of linalool and 70% of nerolidol plus a small amount of benzaldehyde. Unlike niaouli oil which is produced in New Caledonia from *M. viridiflora* Brogn. et Gris, the oil from *M. viridiflora* Sol. contains little or no cineol.

Thyme Oil. The oils of various sub-species of *Thymus serpyllum* found in Switzerland were prepared and analyzed by Hegnauer[130]. The proportions of phenols, citral and esters in the oils were determined and an attempt was made to correlate the analytical values of the oils with the morphological characteristics of the plants.

Tylophora Indica Oil. Rao[131] reported that the roots of *Tylophora indica* (Burm.f.) Merr. contain *p*-methoxy salicylaldehyde. Steam distillation of the alcohol extract of the dried root powder gave 0.26% of crystalline *p*-methoxy salicylaldehyde combined with a small amount of oily material.

Valerian Oil. Wallis and Sanyal[132] determined the essential oil content of various samples of European valerian (*V. officinalis*) and Indian valerian (*V. wallichii*). The distillation apparatus of Meek and Salvin was used with slight modifications. Frothing of the flask contents was controlled by the addition of glycerine and strips of filter paper. The use of mineral oil to reduce frothing was unsatisfactory because it was carried into the receiver with the distillate. A sample of English valerian yielded 1.5% (v/w) of volatile oil while two samples of Belgian valerian yielded 0.70% and 0.76%, respectively. The roots, which made up approximately 68% of the weight of these samples, contained slightly more oil than the rhizomes. Of four samples of commercial valerian of Indian origin, two contained 0.4% (v/w) of volatile oil and the others 0.2% and 0.3%, respectively. The Indian drug consisted mainly of rhizomes, the roots comprising only about 10% of the weight. However the oil content of the roots proved to be double that of the rhizomes, showing that the oil yield of Indian valerian could be raised by including a higher proportion of roots. Valerian roots from plants growing wild in Kashmir yielded 1.0% of essential oil while the rhizomes yielded only 0.4%.

In a study of seasonal variations in the composition of valerian roots, Fauconnet[133] found that the essential oil content of the fresh roots varied between 0.5% and 2.2%. The oil content was at a maximum in February and March and at a minimum in May and June. The oil apparently is formed simultaneously with the reserve substances and the oil content reaches a minimum soon after the reserve materials reach the lowest point late in April. Volatile acids in the fresh roots generally increase when the essential oil content declines and vice versa, but no quantitative relationship between the two constituents was found.

Vanilla. The year 1948 saw Mexico temporarily outrank Madagascar as the world's largest exporter of vanilla. In October of the preceding year, the colonial administration in Madagascar had established compulsory minimum prices for Bourbon vanilla to prevent prices from dropping far below the high point reached during the war. Although there were huge stocks of vanilla stored in Madagascar, minimum prices were set at the extremely high level of $15.00 to $17.00 per kilogram, depending on the quality. Since adequate supplies of Mexican vanilla were available at much lower prices, buyers in the United States turned to Mexico

for their purchases, with the result that exports of vanilla from Madagascar to the United States dropped from 303 metric tons in 1947 to only 23 tons in 1948 (see Table XVII).

In September of 1948, with a large new crop of vanilla in prospect in Madagascar, minimum export prices were lowered to $9.00 to $11.00 per kilogram. Stocks of saleable vanilla beans in storage on the island were estimated at 700 tons. In an effort to stabilize the market, curers were required to destroy a portion of their holdings and the export tax was raised from 21% to 40% ad valorem to provide funds for compensating the curers.

The production of cured vanilla in Madagascar and the Comoro Islands in 1948 amounted to 461 metric tons, of which the Comoros accounted for approximately 50 tons. Large quantities of beans, possibly half of the crop, remained unharvested because of the surplus already on hand.

Table XVII.

Exports of Vanilla from Madagascar[34]

Destination	1946 Metric Tons	1947 Metric Tons	1948 Metric Tons
United States	392	302.9	23.1
France	61.5	39.5	48.8
Other countries	199.5	4.4	4.2
Total	653.0	346.8	76.1

Exports of vanilla from Réunion amounted to 31 metric tons in 1947 and 17 tons in the first six months of 1948. Exports of vanilla from the islands of French Oceania in 1947 totaled 272 metric tons. Of this amount France received 131.2 tons, the United States 71.7 tons and Australia 59.1 tons. During the first six months of 1948 only 55 tons of vanilla were exported from French Oceania.

Before World War II most of the vanilla exported from the French colonies was sent to Marseille for re-export, but in 1946 and 1947 the bulk of the vanilla exported to the United States was shipped direct. Re-exports of vanilla from France to the United States amounted to 51.7 metric tons in 1947 and 42.4 tons in 1948 while re-exports to Great Britain totaled 13.9 metric tons in 1947 and 11.9 tons in 1948.

The high price of vanilla on the world's markets encouraged growers in Mexico to increase their plantings and the 1947-48 harvest was estimated at 551,000 lb. Approximately 60% of the current Mexican output is produced in the district of San José Acateno in the state of Puebla where vanilla culture was first introduced in 1938.

Exports of vanilla from Mexico in 1947 amounted to 485,000 lb. and were valued at $1,112,000. In 1948 vanilla exports rose to 686,000 lb. ($1,407,000) or four times the quantity exported from Madagascar in that year. The average value per pound dropped from $2.29 in 1947 to $2.05 in 1948, a figure far below the minimum price set for the lowest grade of Bourbon vanilla. Most of the Mexican output is sold in the United States.

Vetiver Oil. The island of Réunion exported 10.7 metric tons of vetiver oil in 1947 and 11 tons in 1948. Nearly all of the oil was shipped to France. The wholesale price of vetiver Bourbon on the New York market was in the vicinity of $30 per lb. in January 1948, but with the devaluation of the franc the price dropped considerably. In the last quarter of the year the price ranged from $10 to $12 per lb.

Haiti supplied 22,700 lb. out of the total 33,200 lb. of vetiver oil imported into the United States in 1948. The value of the Haitian vetiver oil was placed at $446,100. The price in the United States was drastically reduced during the year as a result of the drop in price of the Réunion oil.

Vitex-negundo Oil. Basu and Singh[134] distilled an essential oil from the leaves of *Vitex-negundo* Linn. (*Verbenaceae*), an Indian shrub which has medicinal properties. The oil, obtained in yields of 0.01–0.07%, had $n^{20}D$ 1.475, d^{23} 0.9215, acid value 2.7, ester value 24.93 and acetyl value 143.8. The oil was optically inactive and was soluble in about 10 volumes of alcohol. Constituents of the oil included carbonyl compounds (22.5%), phenols (15%) and cineol (10%).

Witch Hazel Oil. Neugebauer[135] showed that the distillate of witch hazel containing approximately 0.015% of the essential oil and no tannin has a hemostatic effect comparable to that of recognized hemostatic preparations. In experiments on rabbits, the intravenous injection of witch hazel distillate shortened bleeding time by one-third and accelerated clotting of the blood. The pure essential oil of witch hazel surprisingly proved less active than the distillate, perhaps due to changes in the composition of the oil during purification. The hemostatic action of the distillate of *Corylus avellana* (hazelnut bush) was about equal to that of witch hazel distillate.

Wormseed Oil. The wormseed crop produced in Maryland in 1947 was poor, the yield of oil being estimated at 25–35 lb. per acre as compared with 60 lb. under the most favorable conditions. Total production amounted to approximately 20 tons in comparison with 56 tons in 1946.

In view of the limitations of current methods for the estimation of ascaridol, Halpern[136] attempted unsuccessfully to devise a new method based on the oxidation of ascorbic acid. Preliminary experiments showed that ascaridol does not react with ascorbic acid in stoichiometric proportions. The reaction of ascaridol with potassium iodide in the Cocking and Hymas procedure does not occur on an equimolecular basis, but there is a fixed relationship between ascaridol and liberated iodine. Halpern[137] investigated the possibility that addition of iodine to the olefinic bond of ascaridol may complicate the reaction. Menthene, terpinolene, limonene and *p*-cymene, when treated with an I_2–KI mixture, failed to absorb iodine even in the presence of ascaridol as catalyst. (Cf. Wagner *et al.*, Anal. Chem. 19: 976, 1947.) Consequently it may be assumed that iodination of the double bond of ascaridol does not occur in the iodometric method of assay.

Cardoso do Vale[138] developed a colorimetric method for determining ascaridol in wormseed oil based on the reaction of ascaridol with ferrous sulfate in hydrochloric acid, which gives a brownish red color. The same author[139] also devised a modification of the Cocking and Hymas determination of ascaridol which avoids the titration of the liberated iodine. The color of the solution due to the presence of iodine is compared with standard solutions made by adding known amounts of potassium dichromate to an acid solution of potassium iodide. The results secured by use of these colorimetric procedures agreed closely with those obtained by the original Cocking and Hymas method.

Wormwood Oil. Fonseca[140] determined the essential oil content of wormwood (*Artemisia absinthium*) grown in Brazil. Two samples of young leaves collected in November contained respectively 0.34% and 0.38% of essential oil on a dry basis while a sample of young leaves gathered in July contained 0.41% of oil. The corresponding values for fully grown leaves were 0.15%, 0.20% and 0.38%, respectively. Fonseca recommends that the Brazilian Pharmacopoeia should specify a minimum volatile oil content of 0.20% for wormwood drug.

Ylang-ylang Oil. Exports of ylang-ylang oil from Madagascar dropped from 56 metric tons in 1946 to 14.8 tons in 1947 and 17.6 tons in 1948. Shipments to the three main importing countries for 1948 were as follows, with 1947 figures in parentheses: France 13.8 (9.0), Great Britain 3.5 (1.5) and United States 0.3 (4.2).

Réunion exported approximately 1 metric ton of ylang-ylang oil in each of the years 1947 and 1948. Exports of the oil from the Comoro Islands in the first half of 1948 totaled 7.5 tons.

AROMATICS

Courtesy of United States Bureau of Plant Industry, Soils, and Agricultural Engineering

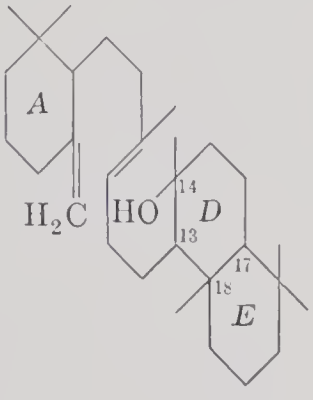
DILL

Notes on Scientific Research in the Field of AROMATICS AND RELATED PRODUCTS

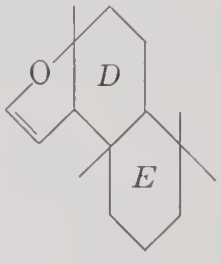
Among the various developments recorded in the literature during 1947 and 1948 and reported in this section there were two of outstanding importance. The first was the synthesis of the irones, *which are substances with an odor of violets occurring naturally in orris root. This accomplishment was made possible by clarification of the structure of the irones, over which there had been much controversy. The second was the utilization of the acyloin condensation to effect ring closure in the synthesis of large ring ketones having an odor of* musk.

Ambrein. The final steps in determining the structure of ambrein, a crystalline triterpene alcohol which occurs in ambergris, were accomplished in 1947 (see Schimmel Report 1946, 83). The structure now accepted is represented by formula I.

Lederer, Mercier and Pérot[141] established that the hydroxyl group is located on carbon 14 as the result of a study of two new products from the oxidation of ambrein: a hydroxyaldehyde $C_{16}H_{28}O_2$ and an unsaturated cyclic oxide formed from it by dehydration. These products have one less carbon atom than ambreinolide, a lactonic oxidation product containing rings D and E. The formation of the unsaturated cyclic oxide requires the hydroxyl group of ambrein to be on carbon 14 rather than 13, the oxide having structure II and ambreinolide the structure of a δ-lactone (III).



I



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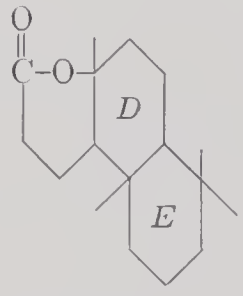


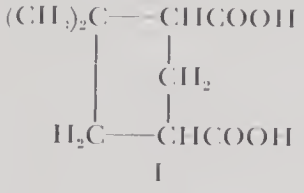
III

The question of the location of the angular methyl group between rings D and E was settled by Jeger, Dürst and Ruzicka[142]. An acid containing these rings which was obtained by the degradation of ambrein was identical with an acid derived from the bicyclic diterpene manool which has a methyl group in the position corresponding to 18.

Apofenchocamphoric Acid. Two methods of synthesizing *cis*-apofenchocamphoric acid (I) from the ethyl ester of 3,3-dimethylcyclopentan-1-one-4-carboxylic acid were reported by Miss Mukherji[143]. In one synthesis the keto-ester was treated with liquid hydrogen cyanide at a low temperature in the presence of potassium cyanide and the crude cyanohydrin thus obtained was digested on the steam bath

with phosphorus oxychloride and pyridine. Hydrolysis of the resulting unsaturated nitrile gave dehydro-apofenchocamphoric acid which was hydrogenated to form *cis*-apofenchocamphoric acid, m.p. 144–145°.

In the second method of synthesis, the ethyl ester of 3,3-dimethylcyclopentan-1-one-4-carboxylic acid was condensed with ethyl cyanoacetate to give ethyl 4,4-dimethyl-3-carbethoxycylopentylidene cyanoacetate. On reduction and acid hydrolysis this was converted to 4,4-dimethyl-3-carboxycyclopentane-1-acetic acid. The lead salt of this dicarboxylic acid on distillation yielded the expected bicyclic ketone, β-isocamphenilone (II), as a colorless liquid, b.p. 197°, having a camphoraceous odor. Oxidation of β-isocamphenilone with potassium permanganate gave *cis*-apofenchocamphoric acid in quantitative yield.



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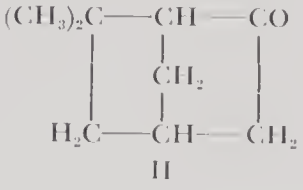
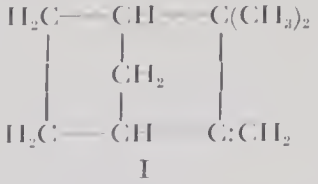


II

Calamene. *See page 9.*

Camphene. Hückel and Kirschner[144] demonstrated that the carbonyl compound (m.p. 31°) which is the main product of the oxidation of inactive camphene (I) by lead tetra-acetate is an R-homocamphenilone. In an earlier paper by Hückel and Hartmann (Ber. 70:959, 1937) this compound was assumed to be camphenilanaldehyde (II). Catalytic hydrogenation of the carbonyl compound gave predominantly an alcohol m.p. 101° (*p*-tolnenesulfonate m.p. 58°) while reduction with alkali yielded a stereoisomer of this alcohol melting at 84° (*p*-toluenesulfonate m.p. 54°). The slow rate of hydrolysis of esters of these alcohols indicated that they were not primary alcohols. On oxidation of the carbonyl compound with selenium dioxide, carbocamphenilone (III) was formed. The diketone might have been derived from either of the two R-homocamphenilones (IV and V) but the strong steric hindrance exhibited by the alcohol of m.p. 101° pointed to structure V in which the carbonyl group is adjacent to the carbon carrying the two methyl groups. R-homocamphenilone (V) is thus the main product of the oxidation of camphene by lead tetra-acetate and it is probably an intermediate in the oxidation of camphene to camphenic acid by means of alkaline permanganate. The alcohols formed on reduction of the ketone are R-homocamphenilols.



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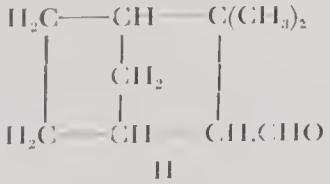

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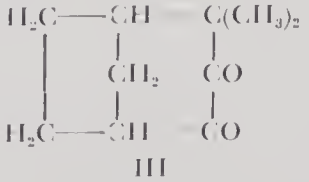
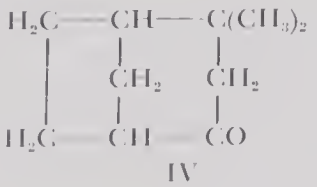
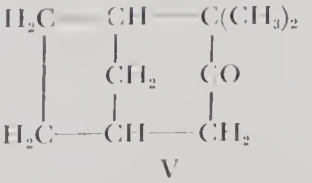


III



IV

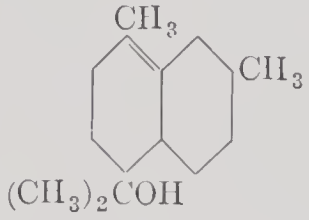


V

Camphor. Angla[145] observed that camphor reacts with thiourea in absolute methanol to form a crystalline molecular complex having the composition $C_{10}H_{16}O \cdot 3CS(NH_2)_2$. The complex is decomposed by water and by most organic solvents.

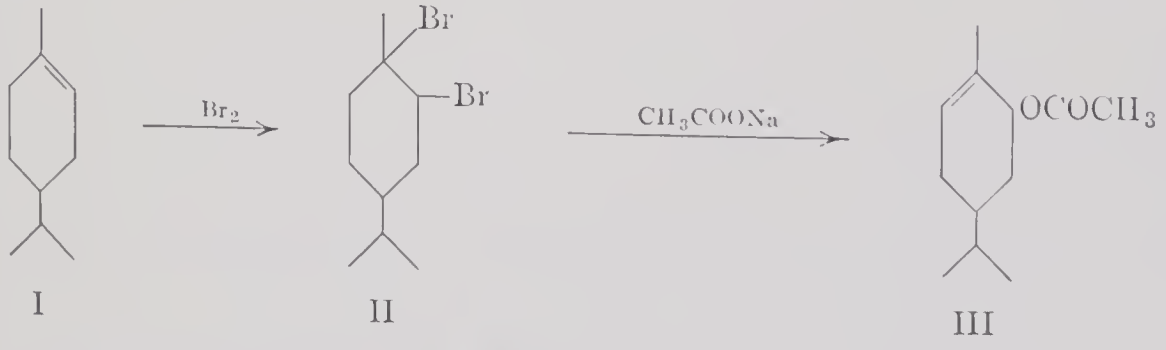
The conversion of camphor oxime to α-campholenonitrile can be effected by means of isothiocyanates, according to Gheorghiu and Budeanu[146]. Camphor oxime treated with phenyl isothiocyanate in alcoholic solution forms an addition product, camphor-O-(anilinothioformyl) oxime, which separates from the solution as a viscous oil. On distillation the addition product is converted to α-campholenonitrile. Treatment of camphor oxime with benzoyl isothiocyanate in benzene also results in the formation of α-campholenonitrile while benzamide and carbon oxysulfide are by-products of the reaction.

Carotol. The constitution of carotol, a sesquiterpenic alcohol found in the essential oil of *Daucus carota* (wild carrot), was investigated by Sorm and Urbánek[147]. On the basis of the products formed on oxidation with potassium permanganate and other reactions the following structure was assigned to carotol:



Hydrogenation of a sample of carotol having $[\alpha]^{18}D + 6.9°$, $n^{20}D$ 1.4997 and d^{20}_4 0.9702 gave dihydrocarotol, a colorless oil distilling at 131–133° at 11 mm.

Carvomenthene. Dupont *et al.*[148] brominated carvomenthene (I) by means of the bromide of pyridine hydrobromide and secured a 50% yield of a liquid dibromide b.p. 135–137° at 12 mm. The reactions of the dibromide, e.g., the formation of carvotanacetyl acetate (III) on treatment with sodium acetate, indicated that it had the structure of 1,2-dibromomenthane (II).

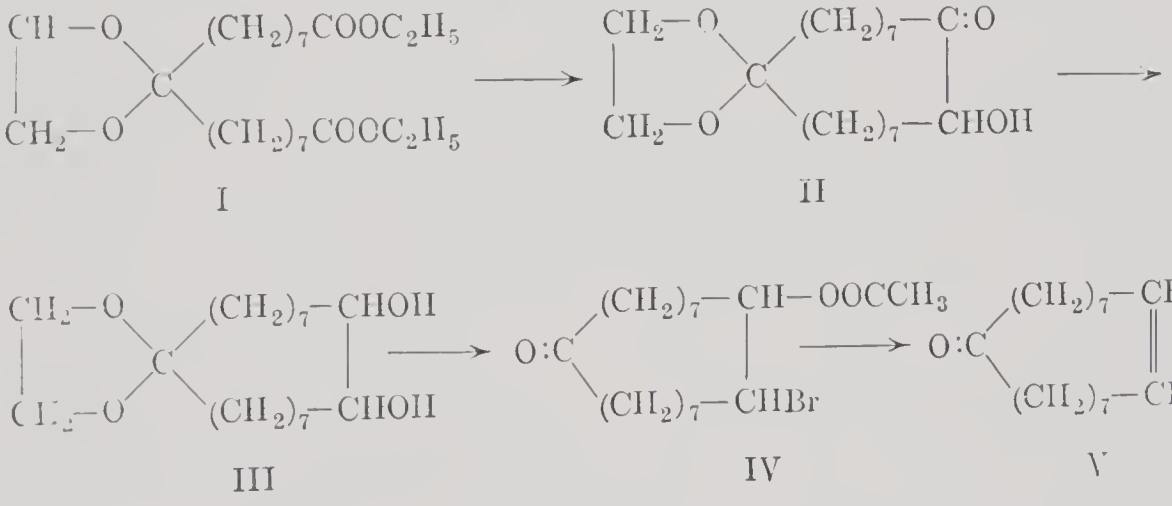


Caryophyllene Oxide. *See page* 18.

Cinnamic Aldehyde Substitutes. In view of the war-time scarcity of cinnamon oil and cinnamic aldehyde (β-phenylacrolein), Bordenca[149] prepared a number of substituted acroleins for testing as sources of the cinnamon odor and flavor. Furanacrolein, a light yellow solid melting at 54°, proved to be very similar to cinnamic aldehyde in taste and odor, although the taste was slightly sweeter. The compound can be made cheaply by the condensation of acetaldehyde with furfural

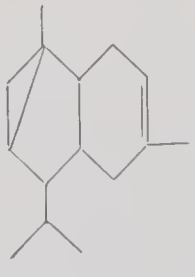
which is produced from waste oat hulls. The taste of the liquid α-methyl derivative of furanacrolein bears little similarity to that of cinnamon. β-Cyclohexenylacrolein (b.p. 80° at 2 mm.) and β-cyclohexylacrolein have a burnt taste and sharp odor. β-Cyclopentenylacrolein (b.p. 73° at 2 mm.) also lacks the cinnamon character.

Civetone. Stoll, Hulstkamp and Rouvé[150] utilized the acyloin condensation for the synthesis of civetone (see page 75). Ethyl-9-ketoheptadecanedioate in the form of its ethylene glycol ketal (I) was cyclized by means of sodium to give the ethylene ketal of 9-keto-1,17-cycloheptadecanolone (II). Catalytic reduction of the acyloin gave the diol (III) which was separated into the α and β isomers. Treatment of the diol with hydrobromic acid in acetic acid converted it to the bromo-acetate (IV) which on reduction with zinc yielded civetone (V). Starting from either the α or β form of the diol, the product obtained was a mixture of α- and β-civetone, semicarbazones m.p. 186–187° and 195–196°, respectively. α-Civetone, identical with natural civetone, was shown to have the *cis* structure and β-civetone the *trans* structure.



Clausenan. *See page* 17.

Copæne. Briggs and Taylor[151] determined the position of the double bond in copæne, a tricyclic sesquiterpene found in the oil of *Phyllocladus trichomanoides*, by the following series of reactions. Treatment of copæne with perbenzoic acid yields an oxide which is converted by methylmagnesium iodide into the corresponding methylcarbinol. On dehydration and dehydrogenation the methylcarbinol is converted to the known compound 1,6,7-trimethyl-4-isopropylnaphthalene. As copæne has methyl groups in positions 1 and 6, the double bond must be in the 6–7 position and the structure of copæne is represented by formula I.



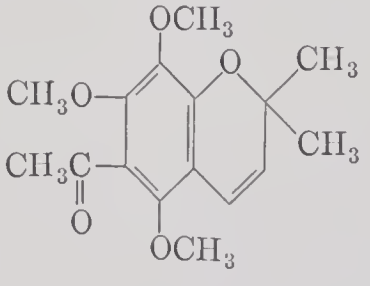
I

Cryptol. The dextrorotatory form of *cis*-cryptol (4-isopropyl cyclohex-2-enol) was isolated for the first time by Gillespie, Macbeth and Mills[152] from the mixture of cryptols obtained by reducing *l*-cryptone with aluminum isopropoxide. Preliminary separation of *d-cis*-cryptol from the main product, *l-trans*-cryptol, was accomplished by recrystallization of the *p*-nitrobenzoates and the *cis* form was purified through the 3,5-dinitrobenzoate, m.p. 113°. *d-Cis*-cryptol was obtained as a fragrant viscous oil, b.p. 70° at 1.5 mm. and $\alpha^{14.5}D$ + 90.2°; *p*-nitrobenzoate m.p. 49°. The two epimers were present in the reaction product in the proportion of 65% *l-trans*-cryptol to 35% *d-cis*-cryptol.

Epoxylinalool. Pigulevskii and Markina[153] carried out the hydrogenation of epoxylinalool which they prepared by the method of Naves and Bachmann (Schimmel Report 1945, 75). The epoxylinalool (b.p. 68° at 8 mm., $[\alpha]D$ + 3.63°) was hydrogenated with a platinum catalyst, giving the corresponding saturated epoxide (b.p. 74° at 10 mm.) in a yield of 80%. The product had d^{20}_4 0.9257, $n^{20}D$ 1.4410, $[\alpha]D$ −4.98° and was completely saturated as shown by the Raman spectrum. The optical rotation was not stable, declining to − 1.17° in four months.

Eugenine and Eugenone. The structures first assigned to eugenine and eugenone, two crystalline compounds occurring in wild cloves from the Moluccas (Schimmel Report 1946, 17) were found by Meijer and Schmid[154, 155] to be incorrect. Further work showed that eugenine (m.p. 119—120°) has the constitution 2-methyl-5-hydroxy-7-methoxy chromone while eugenone (m.p. 97—99°) has the structure 2,4,6-trimethoxy benzoyl acetone. Eugenone represents the first derivative of benzoyl acetone to be found in nature.

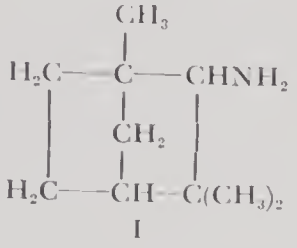
Evodione. Wright[156] reported additional work on the constitution of the crystalline ketone "evodione" which was isolated from the volatile oil of *Evodia Elleryana* (Schimmel Report 1946, 20). The products obtained in degradation experiments could be accounted for on the assumption that evodione has the structure 5,7,8-trimethoxy-6-acetyl-2,2-dimethyl-1,2-benzpyran (I).



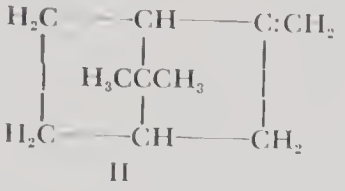
I

Fenchene. The action of nitrous acid on α-fenchyl amine (I), a reaction previously studied by Wallach (Ann. 362: 181, 1908), was further investigated by Hückel[157]. He confirmed the formation of α-fenchene (II) and identified as other products of the reaction the *alpha* and *beta* forms of fenchol, present in approximately equal amounts, and the monocyclic alcohol α-terpineol. The limonene which Wallach reported as one of the reaction products may have been formed by the dehydration of terpineol under the influence of hydrochloric acid, since Hückel, who used acetic instead of hydrochloric acid, could not detect the presence of

limonene. He found no evidence of the formation of a hydrate of α-fenchene to correspond to the formation of camphene hydrate when bornyl amine is treated with nitrous acid.

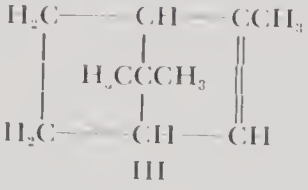


I



II

A new fenchene, designated as ξ-fenchene, was prepared by Hückel and Kindler[158] by treating the *p*-toluenesulfonate of β-fenchol with sodium isopropylate. The hydrocarbon gave *cis*-apocamphoric acid on permanganate oxidation and accordingly was assigned structure III. ξ-Fenchene has the same carbon skeleton as α-fenchene but the double bond is in an endocyclic rather than semicyclic position.



III

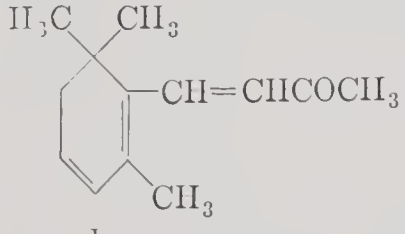
Geraniol. Alquier[159] attempted to prepare citral by the dehydrogenation of geraniol, using a catalyst of reduced copper on pumice stone. The product contained very little citral but apparently consisted of citronellal and other aldehydes formed by the hydrogenation of citral. The catalyst proved satisfactory for the dehydrogenation of saturated alcohols, e.g., for the preparation of isovaleric aldehyde from isoamyl alcohol.

Homovanillin. Challis and Clemo[160] prepared homovanillin (4-hydroxy-3-methoxyphenyl acetaldehyde) in 65% yield by the ozonization of eugenol under special conditions. The hydroxyl group was protected during ozonization by replacing hydrogen with the carbethoxyl group.

Indoles. Buu-Hoï and Royer[161] prepared some of the higher 2,3-substituted indoles using the Fischer method of synthesis which consists in heating the phenylhydrazone of a ketone with anhydrous zinc chloride to effect cyclization. The phenylhydrazones of methyl *n*-hexyl ketone and methyl *n*-nonyl ketone yielded 2-methyl-3-*n*-amyl indole and 2-methyl-3-*n*-octyl indole, respectively. There was no evidence of cyclization involving the methyl group. When ethyl isobutyl ketone was used as the starting material the product was almost exclusively 2-isobutyl skatole. Steric hindrance prevented the methylene group of the isobutyl radical from condensing with the benzene ring. The phenylhydrazone of ethyl hexyl ketone yielded a mixture of 2-*n*-hexyl skatole and 2-ethyl-3-*n*-amyl indole. Other indoles prepared included 2,3-diisopropyl indole from isopropyl isobutyl ketone, 2-phenyl-3-propyl indole from valerophenone and 2-*n*-pentadecyl-3-*n*-tetradecyl indole from palmitone. 2,3-Diisopropyl indole had a balsamic odor but all the other indoles mentioned above had more or less pronounced fecal odors.

Ionones. Naves[162] prepared the semicarbazones (m.p. 157°) and the phenyl semicarbazones (m.p. 181°) of the optically active forms of α-ionone. Hydrogenation of *l*-α-ionone gave a dihydro-ionone the semicarbazone of which melted at 153°. The dihydro derivative of *dl*-α-ionone yields a semicarbazone m.p. 167°. Naves observed that racemic α-ionone has a more intense odor than the optically active forms.

Henbest[163] reported the synthesis of dehydro-β-ionone (I), b.p. 75° at 1 mm., semicarbazone m.p. 144–146°. The substance was prepared by bromination of β-ionone with N-bromosuccinimide and treatment of the brom-substituted product with diethylaniline, but details of the experimental conditions were not published. Karrer and Ochsner[164] obtained different products on treating β-ionone with two mols of bromosuccinimide and heating the bromination products with diethylaniline. One product, which gave a semicarbazone m.p. 182°, was identified as 1-(2',3',6'-trimethylphenyl)-3-butanone (II). A second product was the hydrocarbon Δ^3-dehydro-ionene (III). α-Ionone gave the same products as β-ionone.



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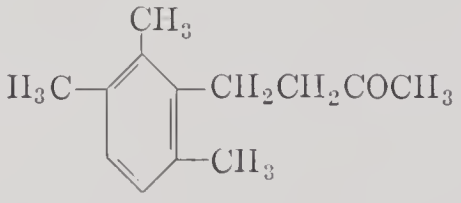

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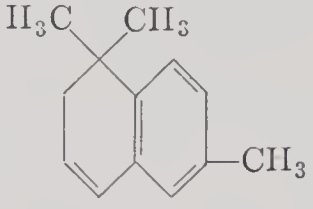
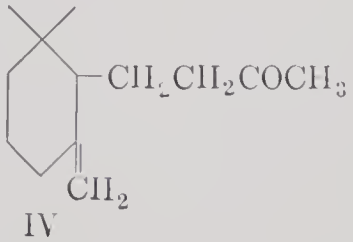
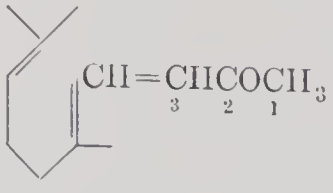


III

Ruzicka *et al.*[165, 166] synthesized dihydro-γ-ionone (IV) by the addition of hydrogen chloride to the ethylenic bond of dihydro-α-ionone and treatment of the product with silver stearate. The semicarbazone (m.p. 188–189°) of the synthetic dihydro-γ-ionone was identical with the semicarbazone of an optically active dihydro-ionone obtained in the oxidative degradation of ambrein (Schimmel Report 1946, 83). Dihydro-γ-ionone was also isolated from the volatile fraction of ambergris. It has the typical amber odor while dihydro-α- and dihydro-β-ionones have a more cedar-like odor.



IV



V

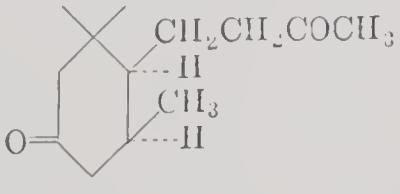
Köster[167] prepared higher homologs of pseudo-ionone (V) by the alkaline condensation of citral with diethyl ketone and with methyl propyl ketone. The former gave 1,3-dimethyl pseudo-ionone while the latter gave a mixture of 1-ethyl pseudo-ionone with a smaller amount of 3-ethyl pseudo-ionone. The branched-chain (3-substituted) products are formed by reaction of the aldehyde group of citral with the α-methylene group of the ketone. Previous workers had overlooked the formation of branched-chain pseudo-ionones from ethyl and propyl ketones due to

their low solubility in sulfite-bisulfite solution. They are easily separated from the normal condensation products by extraction of the bisulfite solution with ether. Examination of technical grades of methyl ionone (from citral and methyl ethyl ketone) revealed the presence of some of the α-methylene condensation product, 3-methyl ionone.

Cyclization of the pseudo-ionones obtained from diethyl ketone and methyl propyl ketone yielded the corresponding α- and β-ionones. The melting points of the respective semicarbazones are given as follows: 1,3-dimethyl-α-ionone 185°, 1,3-dimethyl-β-ionone 164–165°, 3-ethyl-α-ionone 202°, 3-ethyl-β-ionone 182°, 1-ethyl-α-ionone 152–153° and 1-ethyl-β-ionone, 123–124°.

The *trans* modification of tetrahydro-ionone was prepared by Prelog and Frick[168] from a new dihydrocyclocitral, semicarbazone m.p. 206°. Condensation of the aldehyde with acetone gave a crystalline α, β- unsaturated dihydro-ionone, m.p. 50°, which on hydrogenation with a palladium-barium carbonate catalyst yielded a tetrahydro-ionone, semicarbazone m.p. 163°. The previously known tetrahydro-ionone (semicarbazone m.p. 183–184°) was assigned the *cis* configuration and the new isomer the *trans* structure on the basis of their physical properties.

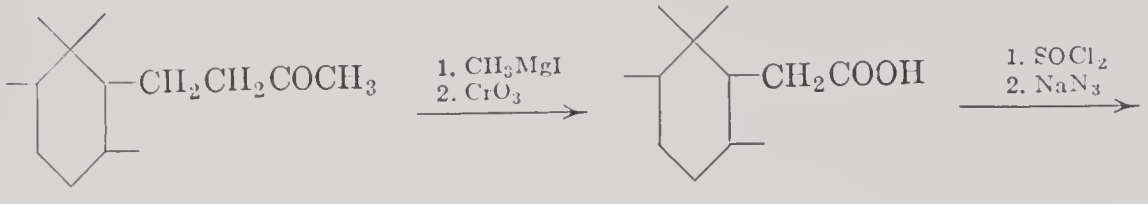
Prelog *et al.*[169, 170] isolated derivatives of ionone from the neutral fraction of the urine of pregnant mares, a by-product of the manufacture of estrone. A number of oxygenated C_{13} compounds were separated, including two isomeric diols (m.p. 110–112° and 121–123°, respectively) which on chromic oxidation yielded the same diketone, bis-(phenylsemicarbazone) m.p. 209–210°. This diketone, which was identical with one isolated from the urine, contained an acetyl group and yielded *cis*-tetrahydroionane on Wolff-Kishner reduction. It was thus recognized as a keto-*cis*-tetrahydro-ionone. The compound did not prove to be identical with any one of three possible isomers (4-oxo-, α'-oxo-*cis*- and β'-oxo-*trans*-tetrahydro-ionone) which were prepared synthetically. By means of degradation reactions the natural diketone was finally identified as 5-oxo-*cis*-tetrahydro-ionone (VI).



VI

A mixture of diol stereoisomers which was isolated by Lederer *et al.*[171] from castoreum gave a diketone on chromic oxidation which proved to be identical with the 5-oxo-*cis*-tetrahydro-ionone isolated from the urine of mares. (See Schimmel Report 1916, 77.)

Irones. As the result of additional studies on the degradation products of tetrahydro-irone and dihydro-irone, Ruzicka[172] and his co-workers abandoned the cycloheptene structure which they had previously attributed to irone. One significant series of degradation reactions involving the side chain of tetrahydro-irone is outlined below.



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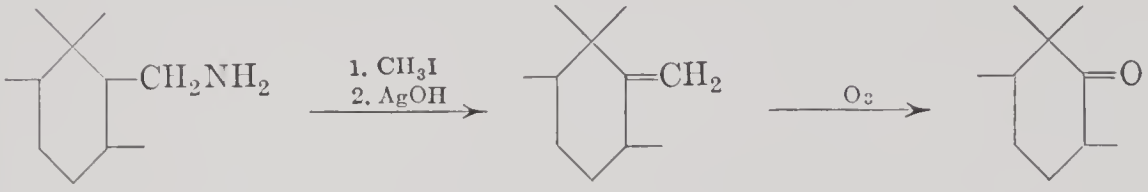


II

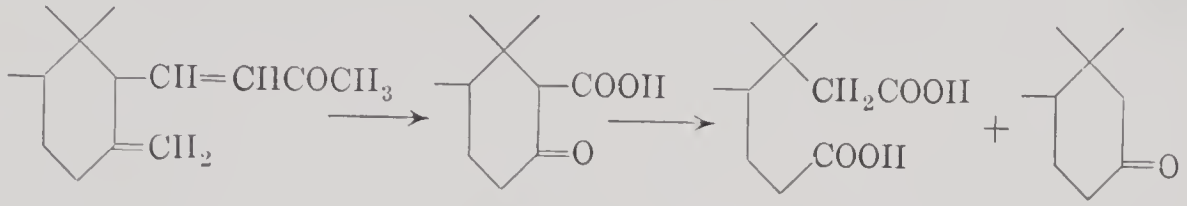
These reactions led to the formation of a ketone $C_{10}H_{18}O$, the semicarbazone of which melted at 214–215°. This ketone proved to be identical with one of two stereoisomeric forms of 1,1,2,5-tetramethyl-6-cyclohexanone (II) which were prepared synthetically by Schäppi and Seidel[173]. This series of reactions established the identity of tetrahydro-irone as 6-methyl-tetrahydro-ionone (I).

Naves[174] found substantial evidence for the 6-membered ring structure of irone in a careful study of its physical properties. An apparently homogeneous sample of natural irone which gave a phenyl semicarbazone m.p. 163° was used in the investigation. On reduction with sodium and alcohol it yielded a dihydro-irol the allophanate of which melted at 162–163°. The physical constants, ultraviolet absorption spectrum and Raman spectrum of the irone and dihydro-irol were determined and compared with the corresponding properties of the ionones, dihydro-ionols and related substances. From the results, Naves concluded that the irone in question had the structure of a 6-methyl-α-ionone.

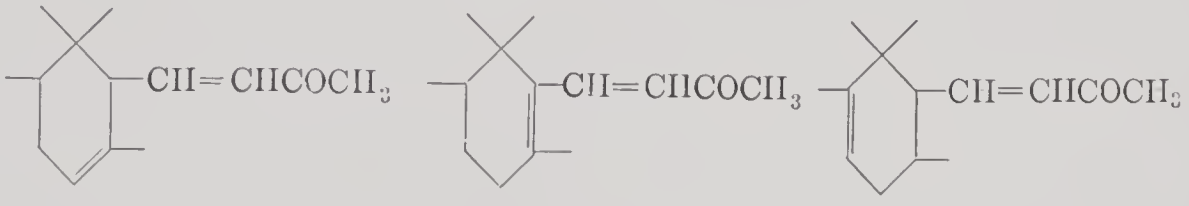
The formation of β,β,γ-trimethyl pimelic acid (IV) on ozonization and chromic oxidation of irone had been considered as a proof of the cycloheptene structure. However, Ruzicka *et al.*[175] realized that this acid might be formed from a 6-methyl ionone having a semicyclic double bond at carbon 3 (formula III). The isolation of a new oxidation product, 2,2,3-trimethyl-6-cyclohexanone (V) confirmed the presence of III, designated as the *gamma* isomer, in natural irone. An irone fraction isolated as the phenyl semicarbazone m.p. 177–179° was recognized as γ-irone.

The regenerated ketone had α_D + 22°, d^{15}_4 0.939 and n^{15}_D 1.505.

Irone which was isolated from orris root without contact with acids was estimated to contain 75% of the *gamma* isomer on the basis of the quantity of formaldehyde formed on ozonization. (Naves,[176] however, considers this estimate to be too high). The ultraviolet spectrum indicated that the remaining fraction consisted of the *alpha* isomer without admixture of β-irone. Weak acids such as oxalic acid cause the isomerization of γ-irone to α-irone (VI) while strong acids or alkalis convert both α- and γ-irone to β-irone (VII). The odor of the *gamma* isomer is slightly inferior to that of α-irone but much finer than that of β-irone.



III IV V



VI VII VIII

Naves[177] prepared a dihydro-γ-irone (semicarbazone m.p. 200°) by the controlled catalytic hydrogenation of *d*-γ-irone isolated from natural irone via the phenyl semicarbazone m.p. 178–179°. The dihydro derivative gave a tetrahydroirone identical with the one obtained from 2,6-*cis*-dihydro-α-irone. The γ-irone in question thus has the 2,6-*cis* structure.

The synthesis of relatively pure α- and β-irone was accomplished by Schinz, Ruzicka, Seidel and Tavel[178] using 2,3-dimethyl-2-hepten-6-one as the starting material. Condensation of this ketone with acetylene in the presence of potassium *t*-butylate and partial hydrogenation of the acetylenic bond gave 3-methyl linalool which by allylic rearrangement was transformed into 3-methyl geraniol. Treatment of 3-methyl geraniol with aluminum isopropylate in acetone-benzene solution yielded 3-methyl pseudo-ionone or pseudo-irone (I), b.p. 95–110° at 0.014 mm.; 2,4-dinitrophenyl hydrazone m.p. 148–150°.

The cyclization of pseudo-irone by means of phosphoric acid yielded *d,l*-α-irone (*d,l*-6-methyl-α-ionone, VI) of d^{15}_4 0.9378 and n^{15}_D 1.4989; phenyl semicarbazone m.p. 166–167°. The ultraviolet absorption spectrum indicated an α-irone content of approximately 95% and the preparation had the typical odor of orris root.

The cyclization of pseudo-irone by means of concentrated sulfuric acid at a temperature of − 5° to 0° gave primarily the *beta* isomer. The *d,l*-β-irone (VII) purified by fractional distillation had d^{15}_4 0.9508 and n^{15}_D 1.5198. Only a portion of the product could be converted to the crystalline phenyl semicarbazone, m.p.

161—166°. This derivative of β-irone, like the phenyl semicarbazone of β-ionone, turns yellow under the influence of light. The corresponding derivatives of α-irone and α-ionone are light-stable.

Naves, Grampololf and Bachmann[179] prepared *dl*-α-irone by the same series of reactions used by Ruzicka and his associates (above) but secured a better yield by a modification of this method in which the methyl linalool was oxidized to methyl citral and the latter condensed with acetone. Cyclization of the pseudo-irone with sulfuric acid gave a *d,l*-α-irone characterized by a phenyl semicarbazone m.p. 175° (cf. the product obtained by Schinz *et al.*). Partial hydrogenation gave a dihydro-α-irone the semicarbazone of which melted at 143—144°. Natural α-irone (phenyl semicarbazone m.p. 162—163°) gave a dihydro derivative of semicarbazone m.p. 173°. From the physical properties of the dihydro-irones, the synthetic α-irone was identified as the 2,6-*trans* isomer and the natural product as the 2,6-*cis* isomer.

By using boron trifluoride as catalyst for the cyclization of pseudo-irone, Naves[180] secured a high yield of *d,l*-2,6-*cis*-α-irone which was purified through the phenyl semicarbazone, m.p. 165°. *Cis*-α-irone has the olfactory properties of natural irone while the *trans* isomer possesses the floral note of the ionones methylated in the side chain. Cyclization with boron trifluoride also yielded a small amount of a new α-irone characterized by a phenyl semicarbazone m.p. 181—182° which is very insoluble in alcohol. This "neo-α-irone" yielded *cis*-dihydro-α-irone of semicarbazone m.p. 173° on hydrogenation with Raney nickel and apparently differed from the 2,6-*cis*-α-irone described above in the spatial arrangement around the ethylenic bond in the side chain. The presence of neo-α-irone in the irone extracted from orris root was demonstrated by Naves[181].

The hydrogenation of *dl-cis*-dihydro-α-irone with Raney nickel and chromic oxidation of the resulting alcohols yielded a tetrahydro-irone regarded by Naves[182] as the 2,3-*cis*,2,6-*cis* isomer. The semicarbazone melted at 199—200°. Hydrogenation of *dl-trans*-dihydro-α-irone yielded approximately 40% of the same tetrahydro-irone and 60% of 2,3-*trans*,2,6-*trans*-tetrahydro-irone, semicarbazone m.p. 161—162°.

The characteristic odor of irone depends upon the presence of the methyl group at carbon 6, as was shown by the synthesis of 4-methyl-α-ionone by Winter, Schinz and Stoll[183] and the synthesis of 5-methyl ionone by Rouvé and Stoll[184]. 4-Methyl-α-ionone had a strong, pure odor of the α-ionone type while 5-methyl ionone (a mixture of the α- and β-isomers) had a weak odor intermediate between the tonalities of ionone and irone.

Naves and Ardizio[185] attempted to synthesize the Δ^5 isomer of irone (VIII) by utilizing the Diels-Alder reaction between 1,1,2-trimethyl butadiene and crotonic aldehyde to form methyl cyclocitral and condensing this aldehyde with acetone.

Courtesy of the Citrus Experiment Station, Reggio Calabria, Italy

HARVESTING JASMINE BLOSSOMS, REGGIO CALABRIA.

ROSE GERANIUM PLANTATION IN SICILY.

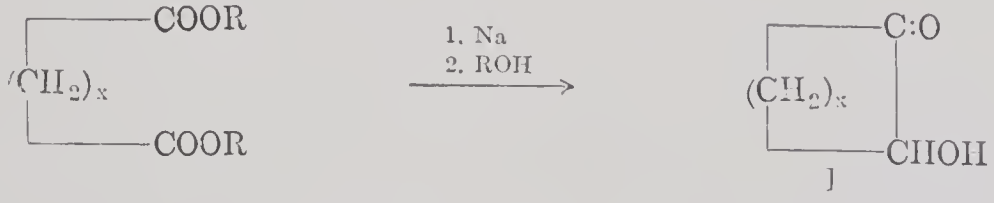
The method yielded only a very small proportion of the desired iso-irone. The main product (phenyl semicarbazone m.p. 171—172°) had the odor of violets although the substance apparently did not have the same molecular skeleton as irone.

Kaurene. The volatile oil of the leaves of kauri (*Agathis australis*) contains a crystalline diterpene m.p. 50° called kaurene (Hosking, 1928). Briggs and Cawley[186] demonstrated that kaurene is identical with podocarprene, a diterpene found in the oil of *Podocarpus macrophylla*, and suggested that the longer name be dropped.

Menthone Oxime. The action of benzoyl isothiocyanate on menthone oxime was investigated by Gheorghiu and Budeanu[187]. When menthone oxime ($[\alpha]^{20}D$ — 16.7°) was treated with benzoyl isothiocyanate in the absence of a solvent, a vigorous reaction took place, resulting in the formation of menthonenonitrile (cf. camphor oxime, p. 65). The reaction when carried out in benzene solution was less violent and gave, in addition to inverted menthone, a 40% yield of 1-(*d*-3-menthen-3-yl)-3-benzoyl-2-thiourea, m.p. 147°, $[\alpha]^{20}D$ + 90°.

Menthyl Oxalate. Naves and Korpi[188] found that the esterification of racemic menthol with oxalic acid yielded two inactive forms of menthyl oxalate melting at 108.5—109° and 106.5—107°, respectively. The higher-melting form was identified by Prelog and Meier[189] as (+)-menthyl-(—)-menthyl oxalate, i.e., the meso form of the ester, and the lower-melting product as the racemic mixture of di-(+)-menthyl oxalate and di-(—)-menthyl oxalate.

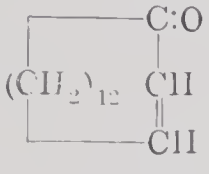
Musk, Synthetic. Hansley's synthesis of cyclic acyloins (U.S. pat. 2,228,268), which provided a new method for the preparation of macrocyclic ketones having a musk odor, was investigated by Stoll[190] and his co-workers. The synthesis consists in treating esters of aliphatic dibasic acids with sodium in boiling xylene, utilizing the dilution principle.



It was found that a high yield of acyloin could be obtained without extreme dilution of the diester providing oxygen were carefully excluded from the reaction mixture. The same observation was also made by Prelog *et al*[191]. Stoll and his associates prepared the series of cyclic acyloins from 1,2-cyclononanolone (formula I, x = 7) to 1,2-cycloöctadecanolone (x = 16) securing yields as high as 85% of the theoretical. Even the 10-membered ring which is usually formed with difficulty was prepared in yields of 30% to 40%. The pure cyclic acyloins are crystalline substances with odors resembling those of the corresponding ketones but somewhat weaker.

Two methods proved satisfactory for converting the cyclic acyloins to the cyclic ketones. One was the direct reduction of the carbinol group by means of zinc and HCl gas in dioxane. The second consisted in dehydrating the acyloin by means of alumina and catalytically reducing the ethylenic bond.

An attempt was made to prepare muskone (β-methyl cyclopentadecanone) from 1,2 cyclopentadecanolone by converting it to 2-cyclopentadecen-1-one (II) by dehydration and treating the unsaturated ketone with methyl magnesium bromide in the presence of cuprous chloride. Only a small proportion of the Grignard reagent added to the conjugated system to yield muskone (III). The main product was the tertiary carbinol (IV) formed by addition of the Grignard reagent to the carbonyl group.



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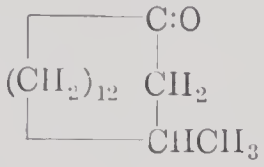

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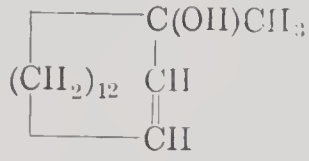
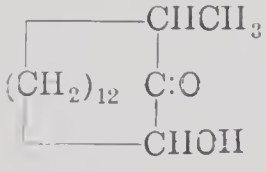


IV

The cyclization of the ethyl ester of 2-methyl pentadecanedioic acid might be expected to yield two acyloins (V and VI). However when the condensation was carried out, only V was obtained. Regardless of the method used to convert the acyloin to a ketone, α-methyl cyclopentadecanone was formed to the exclusion of the β-methyl isomer, muskone.



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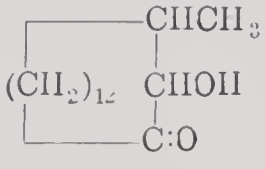
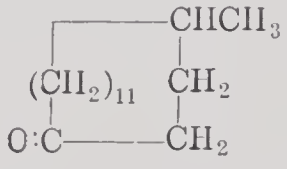


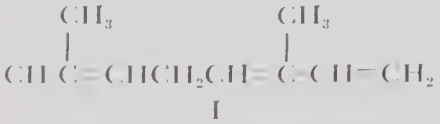
VI



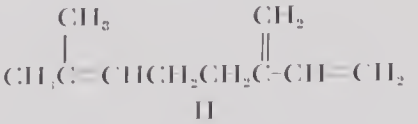
VII

The cyclization of the ethyl ester of 3-methyl pentadecanedioic acid gave both of the two possible acyloins. Reduction with zinc and HCl gas yielded a mixture of muskone (III) and γ-methyl cyclopentadecanone (VII). The odor of the mixed ketones was identical with that of muskone.

Ocimene. Crabalona[192] found that β-ocimene (I) absorbs six atoms of bromine per molecule although two of its three double bonds are conjugated. Myrcene (II), an isomer of β-ocimene which also contains a conjugated system and an isolated double bond, absorbs only four atoms of bromine per molecule. Both terpenes



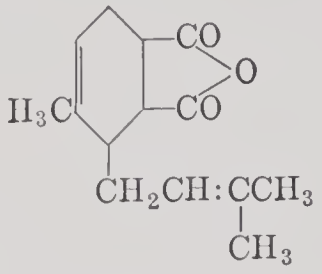
I



II

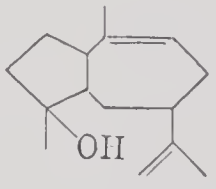
react with maleic anhydride, the addition product from β-ocimene (III) being a liquid, b.p. 128–130° at 2 mm. Hydration of the product converts it into the crystalline dibasic acid, m.p. 163°, which is a useful derivative for the identification

of β-ocimene. By means of this derivative, Crabalona[193] demonstrated the presence of β-ocimene in the terpene fractions of neroli oil and petitgrain oil. The reaction with maleic anhydride is not quantitative and so cannot be used for the estimation of β-ocimene.



III

Partheniol. The resin of the guayule plant, *Parthenium argentatum* Gray, contains the cinnamic acid ester of a sesquiterpene alcohol $C_{15}H_{24}O$ known as partheniol. The structure of this alcohol was determined by Haagen-Smit and Fong[194]. Partheniol crystallizes in long needles melting at 127—128°, is optically active ($[\alpha]^{26}D$ + 116.5° in chloroform, *c* 1.29%) and shows the properties of a tertiary alcohol. Dehydration of partheniol with potassium bisulfate and dehydrogenation of the product with sulfur resulted in the formation of blue S-guaiazulene. The carbon skeleton was thus demonstrated to be identical with that of guaiol and the location of the hydroxyl group and double bonds could be determined by examination of the ozonization products. Accordingly partheniol was assigned the structure represented below.

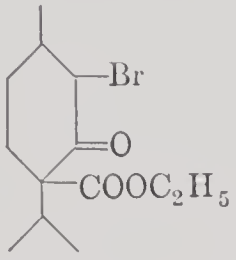


Phellandrene. Berry[195, 196] prepared *l*-β-phellandrene of high purity by debromination of the tetrabromide by means of magnesium in anhydrous ether. A fraction of β-phellandrene purified in this way had a specific rotation of — 74.4°, the highest value yet recorded. The observed molecular refraction of this material was 46.3 whereas the calculated value for β-phellandrene is 45.24. The exaltation is to be expected since the double bonds in β-phellandrene are conjugated.

The debromination of *l*-β-phellandrene tetrabromide with sodium methoxide resulted in removal of only two of the four bromine atoms. Treatment of the product with nascent hydrogen yielded a levorotatory hydrocarbon, $C_{10}H_{18}$, which formed a nitrosochloride melting at 93—94° and having a specific rotation of — 342°. The constants of this derivative indicated that the hydrocarbon was *l*-*p*-menth-1-ene.

Acheson and West[197] reported that the product formed by the reaction of β-naphthol with *l*-β-phellandrene melts at 137—138° and is identical in melting point and optical rotation with the β-naphthol derivative of *l*-α-phellandrene.

Piperitone. Chaudhuri[198] described a new synthesis of *dl*-piperitone. Ethyl-2 isopropyl-5-methylcyclohexan-1-one-2-carboxylate, prepared by the method of Kötz *et al.* (Ann. 357: 256, 1907), on bromination yielded a mono-bromo derivative (I). Heating with quinoline followed by acid hydrolysis converted this compound to *dl*-piperitone (II), b.p. 105—106° at 10 mm.



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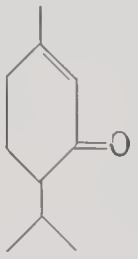
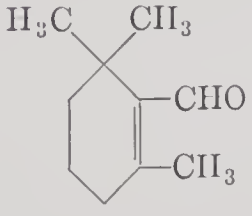


II

Safranal. In an attempt to prepare safranal, Karrer and Ochsner[199] treated β-cyclocitral (I) with N-bromosuccinimide and heated the brominated β-cyclocitral with diethylaniline. The only dehydrogenation product isolated was an isomer of safranal, 2,2,6-trimethyl-3,5-cyclohexadienal (II), semicarbazone m.p. 215° (uncorr.). This new aldehyde which has an odor reminiscent of caraway was also obtained from the bromination product of α-cyclocitral.



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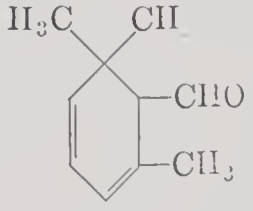

II

Sesquiterpenes. Briggs and Taylor[200] prepared the trinitrotoluene derivatives of cadalene and eudalene. Cadalene trinitrotoluate crystallized in long yellow needles, m.p. 83°, and eudalene trinitrotoluate in short yellow needles, m.p. 62—63°.

MISCELLANEOUS

Courtesy of United States Bureau of Plant Industry, Soils, and Agricultural Engineering

ANGELICA

MISCELLANEOUS

Sources of Essential Oils

The Drug and Oil Plant Division of the California State Department of Education included among its projects in 1917 the solvent extraction of perfume materials from various seeds and plants[57]. Large quantities of celery, carrot and parsley seeds which are unsuitable for planting because of poor germination are available at low cost from California seed companies, providing a potential source of perfumery raw materials. Extraction of celery seed of the Tall Utah variety with isopropyl alcohol yielded 14% of an amber-colored oil. The essential oil of celery seed, separated from the fixed oil by extraction with 95% ethyl alcohol, constituted 22.8% of this product. Carrot seed, on extraction with isopropyl alcohol, yielded 11.5% of an oil containing 7.6% of essential oil. Parsley seed gave a 13% yield of isopropyl alcohol extractive of which the essential oil constituted 10.3%. Caraway seed, obtained from an experimental planting of caraway in the Imperial Valley, on extraction with isopropyl alcohol gave a 20% yield of an oily product containing 18.7% of the essential oil of caraway.

Clary sage plants from a 16-acre experimental planting on the Arroyo Grande Mesa gave a 5.0% yield of the concrete when extracted with isopropyl alcohol. Extraction of clary sage with chloroform gave a 3.2% yield of concrete, the odor of which was excellent. A perfume oil (2.94%) was prepared from the flowers of the everlasting plant (*Helichrysum angustifolium*) by macerating them with mineral oil for four days and treating the mineral oil extract with 90% isopropyl alcohol. Labdanum oleoresin was prepared from young plants of the rock rose (*Cistus ladaniferus*) grown in the Arroyo Grande area. The plants were extracted with chloroform giving a crude labdanum resin which was then dissolved in 95% alcohol. Distillation of the alcohol from the filtered solution left a clear semi-solid oleoresin which melted to a liquid at about 90° F. and had a very pleasing, long-lasting odor. The yield of oleoresin was 5.16% on the basis of the weight of fresh plant material.

Gürgen[201] reported the results of analyses of various essential oils obtained from plants found in Turkey. They are presented in Table XVIII.

Table XVIII.

Properties of Some Essential Oils of Turkey

Plant and Oil Yield*	d^{20}	n	$[\alpha]$	Ester value	Constituents
Salvia officinalis, 1.0%	0.890	1.465	+24.1°	17.2	Borneol and salviol 8.3%
Thymus citriodorus, 0.6%	0.908	1.489	+ 3.1°	29.6	
Thymus squarrosus, 0.3%	0.916	1.501	− 8.8°	11.0	Thymol 60%
Thymbra spicata, 1.3%	0.913	1.495	+ 5°	2.0	Carvacrol 47%
Mentha sylvestris, 0.15%	0.955	1.492	+31.0°	56	Menthol 36.4%
Mentha pulegium var. *pubescens*, 1.2%	0.931	1.475	+33.0°	33.6	Menthol 18.9% pulegone 30%
Nepeta mussunii, 0.1%	1.03	1.485	+20.0°	190.4	
Origanum majorana, 1.7%	0.893	1.467	+25°	36.1	
Satureja calamintha, 0.5%	0.945	1.479	+36.1°	58.8	Pulegone 20%
Echinophora sibthorpiana, 0.7%	0.911	1.489	−10.5°	7	
Echinophora anatolica, 0.05%	0.890	1.476		16.8	
Vitex agnus-castus, 0.75%	0.910	1.477	−12.5°	42.0	

* Based on weight of dry plants

Chopra, Handa and Kapoor[202] tested a variety of aromatic plants growing in Kashmir as sources of essential oils. The yields of oil obtained and the density and refractive index of each oil are given in Table XIX.

Table XIX.

Essential Oils from Plants Growing in Kashmir

Plant	Material Distilled	Oil Yield (%)	d^{15}	n^{20}
Acorus calamus (sweet flag)	Fresh unpeeled rhizomes	3.1	1.08	1.5475
Angelica glauca	Dry roots	13	1.0275	1.529
Elsholtzia cristata	Dry herb	0.93	1.023	1.529
Elsholtzia densa	Dry herb	0.98	0.9697	1.4675
Heracleum Tschemiricum	Dry fruits	0.8	1.0301	1.504
Artemisia dracunculus (estragon)	Flowering shoots	0.7	0.9492	1.5235
Xanthoxylum alatum Roxb.	Dry fruits	2.1	1.025	1.475
Aegle marmelos Corr. (Bengal quince)	Fresh leaves	0.23	0.807	1.448
	Dry leaves	0.54		
Juniperus communis Linn.	Dry berries	0.77	0.9388	1.488
Juniperus macropoda Boiss.	Dry berries	3.3	0.8571	1.473
Ferula jaeschkeana Vatke	Dry roots	1.2	1.119	1.453
	Dry fruits	3.8	0.857	1.461
Archangelica officinalis var. *Himalaica*	Dry roots	0.8	0.8922	1.4478
	Dry fruits	3.8	0.887	1.461
Carum carvi Linn. (caraway)	Seeds (from Baghbanpura)	1.3	0.9095	1.491
	Seeds (from Gurez)	6.8	0.8902	1.486
	Seeds (from Skardu)	8.5	0.8907	1.485
Prangos pabularia Lind.	Dry leaves and flowering tops	0.65 / 1.02	1.129	1.454
	Dry roots	0.76	0.933	1.477
Origanum vulgare Linn.	Herb	1.2	0.9545	1.483
Senecio jacquemontianus Benth.	Roots	0.98	0.9742	1.463
Chaerophyllum villosum Wall.	Whole flowering plant			

Investigations of the essential oil plant resources of Palestine were carried out during the war by the staff of the Industrial Research Institute of the Jewish Agency and the results have been summarized briefly by Halasz and Katz[109]. The yields and properties of the oils from five different wild plants which are available in large quantities are shown in Table XX.

TABLE XX.
PROPERTIES OF OILS OF SOME WILD PLANTS OF PALESTINE

Oil	Laurel (leaf)	*Salvia triloba*	*Satureia thymbra*	*Thymus capitatus*	*Majorana Syriaca*
Yield	0.79%	1.95%	0.6%	0.6%	0.73%
Density	0.918	0.914	0.901	0.918	0.909
Specific rotation		−5.5°	−0.3°		+0.2°
Ester value	27–30	14.6	4.4	4.3	
Solubility in alcohol	2.1 vol. 70%		1 vol. 80%	2 vol. 90%	
Cineol content	76%	75.5%			
Carvacrol content			28–38%	14%	57%[a]
Thymol content				36%	

[a] Total phenols, 72%.

Biogenesis of Essential Oils

In an effort to learn more about the mechanism of perfume formation in plants, Langlais and Bollinger[203] studied the glycosides of rose, jasmine and other flowers. When rose blossoms were soaked in water containing oxalic acid, perfume-forming glycosides were hydrolyzed as shown by the formation of reducing sugars and odorous substances. The sugars were identified as levulose and glucose, undoubtedly present in the form of sucrose. Practically no hydrolysis of the glycosides occurred in water which did not contain acid. The perfume-forming glycosides were extracted from rose blossoms by means of alcohol and then purified; when heated with sulfuric acid solution they gave off an odor of roses and of rum.

It was found that reducing sugars accumulated in neutral infusions of jasmine blossoms, evidence that the glycosides of jasmine, unlike those of the rose, can be decomposed by water without the aid of acid. This difference between the two plants is interesting in view of the fact that jasmine blossoms form additional perfume after they are picked while roses do not. Water-soluble, odorless glycosides were isolated from jasmine blossoms and hydrolyzed by the addition of emulsin. They yielded mannose accompanied by *d*-mannitol and the solution slowly developed a jasmine odor which after eight days resembled the odor of jasmine pomade made by the enfleurage process. The enzymatic hydrolysis of the glycosides was considered too time-consuming to be of interest for the commercial extraction of jasmine perfume. In confirmation of the theories of Charabot, glycosides were found in the green stems and leaves of jasmine, but they did not yield odorous substances on hydrolysis.

According to a patent granted to Brasch[201], the formation of essential oil in cut flowers used for the production of perfume oils can be increased by irradia-

tion of the flowers with electrons. The enzymes which cause wilting and decay are thereby destroyed without affecting the capacity of the plant tissues to synthesize the essential oil. The flowers to be treated are sealed in an airtight container under vacuum and the container, cooled to 0°, is subjected to bombardment by high-speed electrons. After treatment the flowers are stored in a sterile atmosphere for a time to permit the essential oil to accumulate and the oil is then extracted by the customary methods.

Studies relating to the influence of fertilizers on the essential oil content of aromatic plants were reported by Weichan[205]. In field tests on nine different herbs it was found that the application of a balanced fertilizer frequently caused an increase in the size of the plants without having an appreciable effect on the essential oil content. However melissa and marjoram showed some improvement in quality as the result of fertilization.

Methods of Analysis

McKern and Smith-White[206] devised an improved *distillation apparatus* for determining the essential oil content of plant material. The receiver is constructed in such a way that the distillate from the condenser enters from the side and the oil globules which separate from the distillate immediately rise into the graduated neck of the receiver. Since the drops of distillate do not pass through the oil layer there is less opportunity for oil to become mixed with the aqueous phase and return to the distilling flask.

Burdick and Allen[207] developed a *turbidimetric method for determining the amount of peel oil in citrus juices* which is much less time-consuming than the modified Clevenger method. The determination is carried out by distilling a 100-ml. sample of juice with 25 ml. of acetone until 50 ml. of distillate has collected. An aliquot of the distillate is then diluted with two volumes of water and the turbidity of the liquid due to the presence of insoluble essential oil is determined by the use of a photoelectric colorimeter. The optical density can be converted into per cent of recoverable oil by the use of a factor derived by correlating optical density readings with results of peel oil determinations by the Clevenger method.

Concentrated aqueous *solutions of sodium thymotate* are capable of dissolving most oxygenated components of essential oils but will not dissolve terpene hydrocarbons, according to Caujolle and Couturier[208]. Thymotic acid is a substituted salicylic acid and it has long been known that sodium salicylate solutions exhibit the same selective solvent action toward essential oil constituents. A solution of sodium thymotate in water (1:1) acts as a solvent for such oxygenated compounds as camphor, carvacrol, benzaldehyde, carvone, citral, eucalyptol and menthol. Eugenol and other phenols generally are less soluble in the salt solution than are the oxygenated terpene compounds. Sodium thymotate solution may be used to

separate carvacrol from thymol since thymol is less soluble in the solution than carvacrol and crystallizes out first when it is diluted. The solubility of an essential oil in sodium thymotate solution serves as an indication of its terpene content and may reveal adulteration with terpene hydrocarbons.

Hayward and Seymour[209] devised a colorimetric method for determining the *concentration of cedar oil vapor in the air* inside chests made of red cedar wood. The method depends on the reaction of cedrene, one of the constituents of the oil, with vanillin in hydrochloric acid to give a violet color. To carry out the determination, a liter of air is drawn from the chest and bubbled through alcohol which absorbs the cedar oil vapor. An aliquot of the alcohol solution is treated with a 0.1% solution of vanillin in concentrated hydrochloric acid and the concentration of cedrene is determined by colorimetric comparison with standard solutions of cedrene treated with the reagent. The amount of cedrol in the alcohol solution can also be determined by treating another aliquot with phosphoric acid to convert cedrol to cedrene and again determining the cedrene content.

Naves[210] worked out a procedure for the *evaluation of alcohols* in essential oils by the pyridine acetylation method incorporating various improvements on the original method which have been described in the literature.

Gottlieb[211] advocated the use of oxalic acid as dehydrating agent in the *determination of linalool* by the dehydration method.

The gravimetric determination of aldehydes and ketones in the form of their 2,4-dinitrophenylhydrazones was applied by Scholtens[212] to the analysis of a number of essential oils. The reagent was prepared by dissolving 2,4-dinitrophenylhydrazine in concentrated sulfuric acid and diluting with water and alcohol. The gravimetric method gave reproducible results in close agreement with those obtained by other methods when used to determine carvone in caraway oil, carvone in spearmint oil or methyl nonyl ketone in oil of rue. It also gave fairly consistent results in determinations of carvone in dill oil and fenchone in fennel oil for which no reliable methods of assay were previously known. The estimation of benzaldehyde in bitter almond oil and cuminaldehyde in cumin oil by means of 2,4-dinitrophenylhydrazine gave inconsistent results although a modified procedure proved satisfactory for cuminaldehyde.

Lombard[213] examined the *Raman spectra of the terpene oxides* 1,8-cineol (eucalyptol), 1,4-cineol and pinol. The lines which characterize the oxygen-containing tetrahydrofuran and tetrahydropyran rings were missing and the spectra proved to be similar to those of bicyclic terpene hydrocarbons. The Raman line due to the oxide bridge nearly coincided with the line characteristic of the carbon bridge.

Naves and Ardizio[214] determined the *absorption spectra* in the middle ultra-violet region of geraniol, citronellol, "rhodinol", farnesol, linalool, nerolidol, citral, citronellal and farnesal in alcoholic solution. The results were at variance in some particulars with the work of previous investigators. The absorption characteristics of citronellol and "rhodinol" appeared to be identical.

The same author[215] also determined the ultra-violet absorption spectra of various isomeric forms of irone and of α- and β-ionone when dissolved in iso-octane (2,2,4-trimethyl pentane) and compared the results with the spectra of the same compounds in alcoholic solution.

Günthard and Ruzicka[216] analyzed the *infra-red spectra* of the ionones, irones and their hydrogenation products. They concluded that it is not always possible to distinguish between *alpha, beta* and *gamma* isomers in the ionone series on the basis of the infra-red spectra because of the influence of groups in the side chain.

Antibiotic Activity of Essential Oils

The toxic effects of the vapors of various essential oils on one-celled organisms such as paramecium and vorticella were observed microscopically by Svem-Moldavskii[217]. The infusoria, suspended in a water drop above a layer of a volatile oil on a microscope slide, underwent various changes (deformation, bubble formation and disintegration) and were eventually killed by the fumes of the oil. The vapors of lavender, fennel, basil, peppermint and anise oils were lethal to paramecium in ¾, 3, 2, 3, and 25 minutes, respectively.

Piacentini[218] investigated the antiseptic action of bergamot, sweet orange and lemon oils against certain spore-forming bacteria. The oils were tested in the form of aqueous emulsions with sodium oleate as the emulsifying agent. The citrus oils showed strong antiseptic activity against *B. anthracis* and *B. megatherius*, inhibiting the growth of these microörganisms at much lower concentrations than did phenol. It was found that the concentration of oil would have to be at least 1% in order to secure the rapid action required in an antiseptic for practical use. The citrus oils were decidedly less effective against the anaerobic *B. tetani*, with bergamot oil alone showing any superiority over phenol. In the Rideal-Walker test the citrus oils exhibited somewhat weaker germicidal action than phenol against *B. typhi*, a non-sporogen. The phenol coefficient of bergamot and orange oils was 0.41 and of lemon oil, 0.33. No one of the three oils showed a consistent and marked superiority in antibacterial activity over the others in these tests.

Klosa[219] tested the ability of a number of essential oils and essential oil constituents to inhibit the germination of seeds. At a concentration of 1:1000, thyme oil and onion oil completely inhibited the germination of peas while the oils of fennel, peppermint and camomile had no effect. Only 9% germination occurred in the presence of oil of yarrow (1:1000). Thymol at the same concentration entirely

prevented germination while menthol, *l*-borneol, anethol, *d,l*-camphor and fenchone showed no inhibiting activity. The toxic effect of the substances which acted as inhibitors was not permanent as a large proportion of the peas germinated after being washed free of the inhibiting solutions.

Odor Perception

Several papers on the subject of odor perception appeared during 1947 and 1948. Guillot[220] tested the old theory that odorous substances emit particles of some kind other than vaporized molecules which act on the olfactory organs. He attempted to confirm the statement of Durand (1918) that on sudden expansion of air containing water vapor in the presence of camphor, centers of condensation are visible which indicate that particles are given off by the camphor. When this experiment was carried out in the Wilson cloud chamber and repeated using citral, vanillin, menthol and skatole, no evidence was found that any of the odorous compounds gave off particles acting as nuclei for water vapor condensation or that they caused ionization of the surrounding air.

Guillot[221] considers that the odors of substances which some individuals with otherwise normal olfactory perception are unable to smell are fundamental odors involving only one type of olfactory sensation. Such substances include exaltolide and other macrocyclic musks, the steroids found by Ruzicka to have a musk odor, hydrocyanic acid, ambergris, benzyl salicylate, methyl ionone, farnesol and sclareol. It may be noted that ambergris, included in this list, is a natural substance of complex composition and classification of its odor as a fundamental odor is difficult to accept. Guillot[222] found that some individuals are insensitive to the musk odor from one type of compound but can detect the musk odor from another type. This indicates that the odors are actually different although they seem to be identical to people of normal olfactory perception.

Further evidence on this point was provided by Le Magnen[223] in experiments on fatigue of the olfactory organs by compounds of similar odor but different structure. He found that prolonged exposure to the musk odor of exaltolide had little effect on the capacity to perceive the odor of a nitro musk, and vice versa. Hence the two substances must affect different receptor systems.

Analysis of the almond odors of benzonitrile, benzaldehyde and acetophenone by the method of olfactory fatigue showed that, while they have elements in common, they are not identical. After breathing benzonitrile vapor for ten minutes, subjects could detect an odor from benzaldehyde vapor only at high concentrations and the odor was unrecognizable, recalling that of safrole. Only the sharp element in the odor of benzaldehyde was perceptible after inhalation of benzonitrile and safrole simultaneously for ten minutes. In experiments with compounds of camphoraceous odor, olfactory fatigue from hexachlorethane had little effect on sensitivity to the odor of camphor or of borneol but the odor sensation was spicy rather than camphor-like.

Perception of the odor of exaltolide is much more acute in women than in men, according to Le Magnen[224]. Out of 25 men tested, 23 found the odor of

exaltolide imperceptible or weak while only seven out of 25 women so described it. The other 18 women reported the odor as either intense or violent. There was some evidence that sensitivity to this type of musk odor varied in relation to the menstrual cycle, and pre-adolescent girls, with one exception, found the odor of exaltolide to be weak or imperceptible. Thus, sensitivity to this odor seems to be linked to the activity of the female sex hormones.

Le Magnen[225] provided experimental proof that the sensitivity of the olfactory organs to an odor is strongly influenced by the rate at which the scented air is inhaled. Using eucalyptol as the test substance, he found that the minimum concentration in air at which the odor could be detected was doubled when the rate of intake of air was cut from 1600 cc./sec. to 800 cc./sec. The threshold concentration was not influenced by varying the total volume of air breathed in at a constant rate of inhalation. These observations indicate that the degree of stimulation depends on the number of molecules of odorous material reaching the mucous membrane of the olfactory organs in a given interval of time.

STATISTICS

GOLDENROD.

(*See page* 28)

STATISTICAL NOTES

France

The volume of French imports of essential oils during 1947 and 1948 fell short of the total recorded in the pre-war year 1938 by roughly 400 metric tons (see Table XXI). Among the countries listed as the principal suppliers of essential oils to France in 1938, only Italy still ranked as a major supplier in 1948 (Table XXII).

French exports of domestically produced essential oils amounted to only 402 metric tons in 1948 as compared with 1,000 tons in 1938. However, as a result of the rise in prices which occurred during the interval, the 1948 exports of domestically produced oils were worth seven times as much as the 1938 exports. The United States and Great Britain in 1948 were the leading importing countries for essential oils exported from France, as they had been in the last year before the outbreak of World War II (see Table XXIII).

TABLE XXI.

FRENCH PRE-WAR AND POST-WAR TRADE IN ESSENTIAL OILS*[226]

	Quantity in Kg.			Value in Thousand Francs		
	1938	1947	1948	1938	1947	1948
Imports from foreign countries	951,055	567,500	539,518	81,139	628,688	408,758
Imports from French colonies	578,872	437,979	419,125	32,332	376,005	469,990
Exports to foreign countries**	1,101,001	615,031	564,720	167,688	989,067	1,296,213
Re-exports of imported oils	103,364	99,140	162,489	5,666	58,672	121,783

* Excluding terpeneless oils
** Including re-exports

TABLE XXII.

PRINCIPAL SUPPLIERS OF ESSENTIAL OILS IMPORTED INTO FRANCE[226]

	Quantity in Kg.			Value in Thousand Francs		
	1938	1947	1948	1938	1947	1948
Italy	134,401	77,095	50,285	27,485	155,936	101,278
Netherlands Indies	375,828			11,236		
Japan	192,019			11,149		
Bulgaria	6,061	1,041		9,243	74,801	
Indo-China	190,299			8,763		
Madagascar	145,421			5,977		
United States		57,755	15,182		74,948	71,922
India		54,005			59,033	
China			343,825			92,217
Australia			81,923			19,018
Réunion		34,091	30,267		114,476	128,001
Morocco		139,394	102,410		73,931	109,523

TABLE XXIII.

PRINCIPAL CLIENTS FOR ESSENTIAL OILS EXPORTED FROM FRANCE[226]

	Quantity in Kg.			Value in Thousand Francs		
	1938	1947	1948	1938	1947	1948
United States	199,910	119,699	150,569	351,299	269,267	280,982
Great Britain	191,943	71,225	124,121	31,459	177,918	371,842
Switzerland	34,187	61,719	29,789	6,308	52,951	65,067
Germany	79,453			15,051		
Poland	28,444			6,031		
Soviet Union	94,336			5,989		
Netherlands			33,753			63,480
Italy		121,151	11,555		152,600	51,271
Sweden			27,552			66,207
India		49,764			56,691	
Belgium		26,049			11,569	

India

Total exports of essential oils from India for the fiscal years 1947-48 and 1948-49 showed a decided drop in quantity and value in comparison with the exports for the preceding two years (see Table XXIV). A decrease in exports of lemongrass oil was the main factor in the shrinkage of total exports.

TABLE XXIV.

EXPORTS OF ESSENTIAL OILS FROM INDIA[125]

	Gal.	Value in Rupees
1944-45*	131,412	5,358,426
1945-46	168,395	10,033,668
1946-47	151,041	11,631,291
1947-48	101,880	7,856,709
1948-49	111,653	4,691,720

* Year ending March 31.

Italy

Italian exports of essential oils and essential oil terpenes totaled 506 metric tons in 1947 and 521 tons in 1948. While these figures are far below the 1946 total of 734 tons, the volume of exports in 1948 was nearly equal to the volume recorded in the pre-war year 1938. Exports of citrus oils accounted for 472 tons of the 1948 total.

Madagascar

Madagascar's exports of essential oils increased from 444.5 metric tons in 1946 to 576.9 tons in 1947. In the following year the total was 559.7 tons.

TABLE XXV.

EXPORTS OF ESSENTIAL OILS FROM MADAGASCAR[34]

	Quantity in Metric Tons		Value in Thousand Francs	
	1947	1948	1947	1948
France	382.1	429.2	59,776	87,622
Other countries	194.5	130.5	30,518	27,278
TOTAL	576.9	559.7	90,294	114,900

Spain

Spanish exports of essential oils dropped from 380,400 kg. in 1946 to 300,700 kg. in 1947 and 294,000 kg. in 1948. Table XXVI shows the distribution of such exports for the years 1947 and 1948.

TABLE XXVI.

EXPORTS OF ESSENTIAL OILS FROM SPAIN[227]

	Quantity		Value	
Destination	1947 Kg.	1948 Kg.	1947 Pesetas Oro	1948 Pesetas Oro
United States	170,868	101,240	1,528,310	583,588
United Kingdom	34,603	40,948	319,590	375,340
India	3,000	17,938	41,427	150,622
Italy	17,411	38,197	242,417	454,592
Switzerland	16,894	10,179	206,018	92,788
Netherlands	13,397	5,505	223,421	68,623
France		15,777		173,384
Other countries	44,539	64,223	603,680	837,472
TOTAL	300,712	294,007	3,164,563	2,736,409

Switzerland

Exports of essential oils and synthetic perfume materials from Switzerland in 1947 amounted to 260.6 metric tons and were valued at 1,502,000 Swiss francs. Such exports in 1948 totaled 385.5 metric tons and were valued at Fr. 1,740,000.

Imports of essential oils and perfume chemicals into Switzerland dropped from 1,041 metric tons valued at Fr. 17,485,000 in 1947 to 452 metric tons valued at Fr. 5,403,000 in 1948.

TABLE XXVII.

IMPORTS OF ESSENTIAL OILS AND OTHER PERFUME MATERIALS* INTO SWITZERLAND[228]

	Quantity		Value	
Origin	1947 Kg.	1948 Kg.	1947 Fr.	1948 Fr.
United States	278,467	132,551	2,738,458	762,708
India	71,951	29,845	2,201,624	325,725
French East & West Africa	102,783	46,064	1,269,719	63,875
Brazil	27,150	20,105	898,858	442,378
Netherlands East Indies	50,009	10,718	2,206,443	113,077
France	36,992	31,779	1,478,307	949,343
Great Britain	15,244	4,817	382,034	113,225
Spain	18,796	13,508	326,211	171,339
Italy	19,082	8,616	851,678	314,367
China	211,824	25,510	2,342,893	293,376
Other countries	208,602	128,805	2,788,658	1,430,608
TOTAL	1,040,900	452,321	17,484,883	5,403,487

* Fruit esters, camphor, menthol, etc.

United Kingdom

United Kingdom exports of natural essential oils which were produced in that country amounted to 173,251 lb. (£232,168) in 1947 and 339,108 lb. (£436,949) in 1948. United Kingdom re-exports of natural essential oils imported from other countries totaled 214,185 lb. (£191,403) in 1947 and 204,645 lb. (£170,627) in 1948.

Table XXVIII shows the quantity and value of natural essential oils imported into the United Kingdom during the years 1947 and 1948. In 1947, United Kingdom imports of synthetic essential oils from France totaled 7,354 lb. (£72,443) and from Switzerland, 17,168 lb. (£121,035). Imports of floral concretes from France during the same year reached a total of 3,836 lb. and were valued at £112,815.

TABLE XXVIII.

IMPORTS OF ESSENTIAL OILS INTO THE UNITED KINGDOM[229]

Origin	Quantity 1947 Lb.	Quantity 1948 Lb.	Value 1947 £	Value 1948 £
India	194,294	170,323	190,101	151,869
Ceylon	163,862	142,622	58,027	76,288
Other British countries	1,339,826[a]	1,097,638	456,535	344,396
Netherlands	21,939	109,729	23,656	169,406
France	208,767	430,689	291,227	361,161
Italy	680,863	410,631	714,587	469,028
Palestine	38,063	76,958	25,979	44,710
Other countries	998,185[b]	458,470	957,440	374,625
TOTAL	3,645,799	3,197,060	2,717,552	1,994,183

[a] Includes 438,605 lb. from Zanzibar and Pemba, 465,385 lb. from Australia, and 225,572 lb. from Hongkong.

[b] Includes 348,437 lb. from Brazil.

United States of America

Statistics on exports of essential oils from the United States in 1947 and 1948 are presented in Tables XXIX and XXX. It will be noted that exports of unblended essential oils to Canada, the principal market, were nearly identical for these two years while shipments to India, the United Kingdom and Australia were much larger in 1947 than in 1948. The official figure for exports of essential oils to Cuba in 1948 is extraordinarily high and would appear to be the result of an error in tabulation or classification of exports. Citrus oils represented 340,660 lb. of the total United States exports of essential oils in 1947 and 201,260 lb. of the total exports in 1948.

TABLE XXIX.

EXPORTS OF ESSENTIAL OILS FROM THE UNITED STATES[106]

Destination	Quantity 1947 Lb.	Quantity 1948 Lb.	Value 1947 $	Value 1948 $
Canada	290,569	290,903	1,256,923	1,260,055
India	239,721	64,338	785,459	163,991
United Kingdom	218,126	49,324	1,531,210	325,646
Netherlands	171,894	147,465	502,621	519,743
Australia	131,882	30,032	612,369	78,641
France	79,121	88,204	444,625	306,105
Brazil	85,303	74,014	392,642	404,323
Venezuela	82,223	20,843	95,949	92,873
Jamaica	80,259	190	27,049	1,325
Cuba	98,306	953,619	387,604	557,123
Mexico	76,207	74,596	356,101	301,957
Argentina	70,241	31,190	262,298	82,757
Switzerland	62,195	19,596	144,988	60,893
Curacao	33,704	59,381	15,598	23,983
Philippines	27,277	57,130	66,760	101,948
Union of South Africa	55,453	39,683	182,412	173,552
Sweden	47,476	3,607	208,901	7,288
New Zealand	43,515	2,104	164,122	10,857
Other countries	297,607	367,933	926,795	673,790
TOTAL	2,191,079	2,374,152	8,364,426	5,146,850

TABLE XXX.

EXPORTS OF ESSENTIAL OIL MIXTURES FROM THE UNITED STATES[106]

Destination	Quantity 1947 Lb.	Quantity 1948 Lb.	Value 1947 $	Value 1948 $
Mexico	151,741	144,146	641,699	561,707
Cuba	125,022	125,577	773,389	650,296
Canada	84,249	56,356	713,868	260,649
Colombia	12,775	27,959	331,585	187,908
Venezuela	22,110	23,635	153,215	150,722
Brazil	84,235	49,446	714,175	378,108
Argentina	66,191	34,242	628,970	232,329
India	73,017	18,545	344,275	101,715
Australia	37,377	4,032	278,317	37,662
Union of South Africa	26,167	39,312	163,195	152,893
Chile	11,245	21,865	104,903	121,777
Philippines	16,489	20,106	80,546	88,174
China	7,761	22,800	22,281	66,364
Other countries	95,627	44,121	526,057	256,903
TOTAL	817,006	632,142	5,476,475	3,247,207

The volume of imports of essential oils into the United States reached 8,000,000 lb. in 1946, but was considerably smaller in 1947 and 1948, as shown in Table XXXI. United States imports of ambergris, civet and musk for the year 1947 were valued at $187,000 while imports of these fixatives for 1948 were valued at $125,000. Imports of floral absolutes and concretes are shown in Table XXXII.

Table XXXI.

Imports of Essential Oils into the United States[230]

Origin	Quantity 1947 Lb.	Quantity 1948 Lb.	Value 1947 $	Value 1948 $
Brazil	1,191,591	887,037	1,959,068	687,983
India	110,651	222,239	903,817	348,977
Australia	199,631	521,614	386,839	307,797
China	756,300	510,831	805,572	414,010
Spain	376,844	261,692	184,809	232,290
Madagascar	273,563	228,680	780,405	448,062
Guatemala	382,385	791,572	921,242	810,821
Paraguay	219,649	302,611	630,769	722,109
France	213,866	151,937	1,605,005	1,135,785
Ceylon	530,238	367,086	715,330	244,994
Italy	94,195	391,456	336,455	1,050,759
Mexico	108,111	116,128	108,993	114,359
Honduras	76,653	66,637	213,031	72,225
Haiti	78,766	171,517	308,627	690,418
Soviet Union	28,841	13,986	73,239	286,455
Netherlands Indies	107,161	569,156	657,433	549,520
Japan	15,000	257,800	22,743	84,032
Other countries	356,795	386,924	1,718,683	1,129,210
Total	6,113,246	6,519,203	12,962,063	9,629,851

Table XXXII.

Imports of Floral Absolutes and Concretes into the United States[230]

Origin	Quantity 1947 Lb.	Quantity 1948 Lb.	Value 1947 $	Value 1948 $
France	1,993	1,611	887,012	375,276
Italy	102	1,838	279,806	232,927
Egypt	278		201,980	
Other countries	192	166	87,155	23,135
Total	2,865	3,618	1,458,983	631,338

BIBLIOGRAPHY

Bibliography

1. Navis, Yves-Rinl. Recent progress in the knowledge of essential oils. Perfumery Essent. Oil Record 39: 315–46. 1948.

2. Smith, J. L. B., and Roux, D. G. The essential oil of *Agathosma gnidioides* Schlechter. Trans. Roy. Soc. S. Africa 31: 333–45. 1917.

3. Ramstad, E. The volatile oil of angelica leaves. Medd. Norsk Farm. Selsk. 9 (9): 175–77. 1947.

4. Mori, D. The determination of anethole in oil of anise. Riv. ital. essenze, profumi, piante offic., olii vegetali, saponi 29: 360. 1947.

5. Briggs, L. H. and Taylor, W. I. The occurrence of isophyllocladene in the essential oil of *Araucaria excelsa*. J. Soc. Chem. Ind. 66: 168. 1947.

6. Gol'mov, V. P., Trofimov, V. I. and Afanas'ev, N. M. Composition of the oil of *Artemisia austriaca*. J. Gen. Chem. (U.S.S.R.) 18: 175–78. 1948.

7. Warriner, C. E. Report on the laboratory work carried out by the drug and oil plant project. Proc. Confer. Cultivation Drug Assoc. Econ. Plants in Cal. 1947, 191–208.

8. Parihar, D. B. and Dutt, S. Chemical examination of *Artemisia scoparia* Waldst. and Kit. Isolation of an essential oil and lactone. Proc. Indian Acad. Sci. 25A: 153–58. 1947. Ibid. 26A: 93–96. 1947.

9. Briggs, L. H. and Taylor, W. I. The occurrence of methyl salicylate in a fern, *Asplenium lamprophyllum*. Trans. Proc. Roy. Soc. New Zealand 76 (4): 597. 1947.

10. Finley, W. H. and Warriner, C. E. California sweet basil oil. Proc. Confer. Cultivation Drug Assoc. Econ. Plants in Cal., 1947, 214–17.

11. United States. Deparment of Agriculture. Report of the Federal Experiment Station in Puerto Rico. 1946. Washington, D. C., Nov. 1947. 55 pp.

12. Giuffre, M. Production and exportation of the essential oil of bergamot. Ind. parfum. 4 (9): 357. 1949.

13. Albricci, A. Characteristics of the bergamot oil of the 1947–48 season. Boll. uffic. staz. sper. ind. essenze e deriv. agrumi 18 (1): 13–16. 1948.

14. La Face, F. The *pelatrice* machine in the bergamot industry. Boll. uffic. staz. sper. ind. essenze e deriv. agrumi 18 (1): 3–9. 1948.

15. Oppenheim, M. Local sensitization of skin to grenz rays by bergamot oil. J. Investigative Dermatol. 8 (5): 255–62. 1947.

16. Naves, Y.-R. Volatile plant materials. 57. Ketones of the essential oil of cabreuva (*Myrocarpus frondosus* and *M. fastigiatus* Allem.) and of bois de rose (*Aniba rosaedora* Ducke). Helv. Chim. Acta 31: 44–49. 1948.

17. Penfold, A. R. and Morrison, F. R. The essential oil of a physiological form of *Boronia ledifolia* (Gay). J. Proc. Roy. Soc. N. S. Wales 82: 71–71. 1948.

18. Naves, Y.-R. and Parry, G. R. Volatile plant materials. 45. Presence of ionones in the concrete essence of *Boronia megastigma* Nees. Helv. Chim. Acta 30: 419–22. 1947.

19. Fester, G. A. Essential oils of indigenous plants of the Republic of Argentina and the surrounding area. Ciencia e invest. Buenos Aires 3: 237–40. 1947.

20. Naves, Y.-R. Volatile plant materials. 43–44. Presence of nerolidol in essential oils of the *Papilionaceae*. Helv. Chim. Acta 30: 275–76. 1947.

21. Sorm, F. and Herout, V. Terpenes. 5. The terpenic constituents of the essential oil of sweetflag (*Acorus calamus* L.). Collection trav. chim. tchécoslov. 13: 177–205. 1948.

22. Anonymous. Output of camphor products, Japan. Drug & Cosmetic Ind. 63 (5): 675. 1948. Mfg. Chemist 21 (3): 138. 1950.

23. KHOTIN, A. A. Fruit formation and accumulation of essential oils in caraway. Doklady Vsesoyuz Akad. Sel'sko-khos. Nauk im. V. I. Lenina 13(12): 17–21. 1948.

24. BAILEY, L. F. Leaf oils from Tenessee Valley conifers. J. Forestry 16: 882–89. 1918.

25. CEYLON, COMMERCE AND INDUSTRIES DEPARTMENT. The Ceylon Trade Journal 14(2): 111. 1949.

26. FUCHS, L. Determination of cinnamaldehyde in oil of cinnamon by means of hydrazine sulfate. Scientia Pharm. 16: 50–56. 1948.

27. GREENE, L. W. Chemical microscopy of essential oils. I. Cassia and cinnamon oils. Am. J. Pharm. 119: 59–65. 1947.

28. WIT, F. Citronella grasses of Ceylon and Java. Landbouw 20: 97–106. 1948.

29. KEIL, H. Contact dermatitis due to oil of citronella. J. Investigative Dermatol. 8(6): 327–34. 1947.

30. WATERHOUSE, D. F. Physiology and toxicology of blowflies. 13. Insectary tests of repellents for the Australian sheep blowfly *Lucilia cuprina*. Australia Council Sci. Ind. Research Bull. 218: 19–29. 1947.

31. MEIJER, T. M. The essential oil from the leaves of *Clausena anisata* Hook f. Rec. trav. chim. 66: 395–100. 1947.

32. NARASIMHA RAO, P. L. Constitution of α-clausenan. J. Sci. Ind. Research (India) 7B(1): 11–12. 1948.

33. ZANZIBAR CLOVE GROWERS ASSOCIATION. Quarterly Market Reviews. 1947–48.

34. MADAGASCAR. DIRECTION DES DOUANES. Statistiques du commerce et de la navigation. 1948. Tananarive, 1949.

35. TREIBS, W. Caryophyllene oxide, its preparation by autoxidation of caryophyllene and its occurrence in plant oils. Chem. Ber. 80: 56–63. 1947.

36. NAVES, Y.-R. Studies on the volatile plant materials. 60. Presence of epoxy-dihydrocaryophyllene (caryophyllene oxide of Treibs) in the benzene extract of crushed clove buds. Helv. Chim. Acta 31: 378–81. 1948.

37. BARTELS, H. A. The effect of eugenol and oil of cloves on the growth of microorganisms. Am. J. Orthodontics Oral Surg. 33²: 158–65. 1947.

38. BASU, N. K. AND DANDIYA, P. C. Chemical investigation of *Convolvulus pluricaulis* Chois. J. Am. Pharm. Assoc., Sci. Ed. 37: 27–28. 1948.

39. SUTHERLAND, M. D. The odor of *Coprosma foetidissima*. New Zealand J. Sci. Technol. B. 29: 91–95. 1947.

40. INDIA. FOREST RESEARCH INSTITUTE. Forest Research in India and Burma. 1915–16, 83–84.

41. CRABALONA, L. Crystalline lactone in oil of costus root. Bull. soc. chim. France, mem. 1948, 357–58.

42. NAVES, Y.-R. Studies on the volatile plant materials. 65. On the lactone $C_{15}H_{18}O_2$ isolated by Ukita from costus root. Helv. Chim. Acta 31: 1172-76. 1948.

43. TREIBS, W. Agropyrene, a natural aromatic enyne hydrocarbon of the couch-grass root. Chem. Ber. 80: 97–101. 1947.

44. THURIAUX, L. A new essential oil extracted from *Cymbopogon densiflorus*. Inst. Roy. Colon. Belge B. des Seances 18: 348-53. 1947.

45. NEUHAUS, J. W. G. AND REUTER, F. H. A note on some minor constituents of the wood oil of *Callitris glauca*, R. Br. Australian Chem. Inst. J. & Proc. 15: 185–89. 1948.

46. BRIGGS, L. H. AND TAYLOR, W. I. The essential oil of *Dacrydium kirkii*. J. Org. Chem. 12: 551–57. 1947.

47. Australia, Bureau of Census and Statistics. Australian statistics of overseas imports and exports and customs and excise revenue. 1946-47. Melbourne.

48. Ralph, C. Minor constituents of eucalyptus oils. J. Proc. Roy. Soc. N. S. Wales 80: 208-11. 1947.

49. Watson, E. M. and Gardner, C. A. Essential oils of the western Australian eucalypts. 8. Oils of *Eucalyptus campaspe* and *E. kochii*. J. Roy. Soc. W. Australia 31: 33-35. 1944-45. (Pub. 1948).

50. Penfold, A. R., Morrison, F. R. and McKern, H. Studies in the *Myrtaceae* and their essential oils. 2. Some sources of error in the study of plant populations: *Eucalyptus citriodora* Hook. Researches on Essent. Oils Australian Flora 1(1): 5-7. 1948.

51. Penfold, A. R. and Morrison, F. R. The occurrence of a physiological form of *Eucalyptus citriodora* Hooker. Australian J. Sci. 11(1): 29. 1948.

52. Berry, P. A. Field observations of the seasonal variations in the yields of the essential oil of *Eucalyptus cneorifolia*. Australian Chem. Inst. J. & Proc. 14: 173-75. 1947. The seasonal variation of the essential oil from the growing tips of *E. cneorifolia* with special reference to the occurrence of cymene. Ibid. 176-200. The action of sodium sulfite on cryptone. Ibid. 201-6. Examination of the terpenes contained in the oil of *Eucalyptus cneorifolia* distilled at the peak of the flush period. Ibid. 383-86.

53. Schmidt, H. High boiling fractions of Spanish eucalyptus oil from *E. globulus*. Chem. Ber. 80: 528-33. 1947. 3-Isopropylidene-1-acetyl-cyclopentene-5, a new constituent of Spanish eucalyptus oil from *E. globulus*. Ibid. 533-38.

54. Jacobson, M. and Haller, H. L. The insecticidal component of *Eugenia H.* identified as 1,8-cineol. J. Am. Chem. Soc. 69: 709-10. 1947.

55. Kir'yalov, N. P. Discovery of dextrorotatory β-pinene in the essential oil of fruits of *Ferula foliosa* Lipsky. J. Appl. Chem. (U.S.S.R.) 20: 1304-7. 1947.

56. Kir'yalov, N. P. Differences in the composition of essential oil of the fruits and roots of *Ferula jaeschkeana* Vatke. Sovet. Botan. 15: 356-57. 1947.

57. Kidder, M. C. Summary of the progress of the drug and oil plant project. Proc. Confer. Cultivation Drug Assoc. Econ. Plants in Cal. 1947. 12-81.

58. Grekhnev, M. V. Essential oils of the fir tree and possibility of their industrial production in Komi, Armenian Republic. Lesnaya Prom. 1947 (2), 15-18.

59. Stoll, A. and Seibeck, E. Allium compounds. 1. Alliin, the true mother compound of garlic oil. Helv. Chim. Acta 31: 189-210. 1948.

60. Holland, B. R., Johnson, P. R. and Sorrells, J. H. Essential oil production in Texas. 2. Sweet goldenrod. Bull. Agr. Mech. Coll. Texas Ser. 5, vol. 1, no. 9. Sept. 1, 1948. 22 pp.

61. Sabulay, S. and Halasz, A. Palestine grapefruit neroli and petitgrain oils. Ind. parfum. 2:200. 1947.

62. Islip, H. T. and Major, F. Grapefruit oil from Palestine. Bull. Imp. Inst. 16(2-4): 215-18. 1948.

63. Kesterson, J. W. and McDuff, O. R. Florida citrus oils; commercial production methods and properties of essential oils (1947-48 season). Florida Agr. Expt. Station. Bull. 452. Gainesville, Fla. Nov. 1948. 11 pp. Illus.

64. Crabalona, L. and Teisseire, P. *d*-α-Pinene in concrete of immortelle de Provence. Bull. soc. chim. France, mem. 1948, 270-71.

65. Stoll, A. and Seibeck, E. Isolation of sinigrin as the genuine crystalline parent substance of horseradish oils. Helv. Chim. Acta 31: 1432-34. 1948.

66. Delepine, M. and Badoche, M. A pinocamphone derived from hydroxy-2-pinanone-3 (from *d*-pinene). Ann. Chim. 3: 573-84. 1948. *d*-Iso- (or *cis*) pinocamphone. Its semicarbazone. Ibid. 585-90. Pinocamphone of hyssop oil. Ibid. 591-93.

67. La Face, D. Yield and characteristics of the oil of jasmine as a function of the season and hour of collection. Boll. uffic. staz. sper. ind. essenze e deriv. agrumi 18(2): 3–11. 1948.

68. Kurth, E. F. and Lackey, H. B. The constituents of Sierra juniper wood (*Juniperus occidentalis*, Hooker). J. Am. Chem. Soc. 70: 2206–9. 1948.

69. Kir'yalov, N. P. Essential oil of *Ledum palustre*. Doklady Akad. Nauk S.S.S.R. 61: 305–8. 1948.

70. Igolen, Georges. Lavandin oil. Parfumerie moderne 40(10): 35–42. 1948.

71. Costa, A. F. and Cardoso do Vale, J. Essential oil of *Lavandula Stoechas* L. Comparison with the oil of *L. pedunculata* Cav. Noticias Farm. (Port.) 1948. 13 pp.

72. Caujolle, F., Coutturier, P. and Bergal, G. The lavenders of Quercy. Parfumerie moderne 40(10): 43–45. 1948.

73. Benezet, L. Lavender oil. Parfumerie moderne 40(10): 31–34. 1948.

74. La Face, D. Influence of some factors in the production of lavender oil. Boll. uffic. staz. sper. ind. essenze e deriv. agrumi 18: 17–23. 1948.

75. Coomber, H. E. and Cosgrove, D. J. Lemongrass oil from Tanganyika. Bull. Imp. Inst. 45(1): 3–6. 1947.

76. a). Bull. Imp. Inst. 46(2–4): 166–67. 1948. b). Jamaica. Department of Agriculture. Annual report for the years ending March 1947 and March 1948. Jamaica.

77. Naves, Y.-R. Volatile plant materials. 56. Some new ketones, the tagetenones, isolated from the essential oil of *Lippia asperifolia*. Helv. Chim. Acta 31: 29–32. 1948.

78. Cosgrove, D. J., Islip, H. T. and Thain, E. M. Reports of recent investigations at the Imperial Institute. Oil of *Melaleuca bracteata* from Kenya. Bull. Imp. Inst. 46(1): 46–50. 1948.

79. Mitchell, W. Reports of recent investigations at the Imperial Institute. Oil of *Melaleuca bracteata* from Kenya. Bull. Imp. Inst. 46(1): 50–53. 1948.

80. United States. Department of Commerce. Foreign Commerce Weekly 37(9): 29. Nov. 28, 1949.

81. Simmons, W. H. Some analytical data on the essential oils of Brazil. Perfumery Essent. Oil Record 38: 264–66. 1947.

82. Naves, Y.-R. Volatile plant materials. 63. Essential oil of *Micromeria abyssinica* (Hochst.) Benth., source of *d*-isomenthone. Helv. Chim. Acta 31: 932–36. 1948.

83. Sabetay, S., Igolen, G. and Monod, C. On the phenols of the concrete essence of mignonette, (*Reseda Odorata*). Ind. parfum. 3: 85–86. 1948.

84. Cavicchi, G. Determination of allyl isothiocyanate. Riv. ital. essenze, profumi, piante offic., olii vegetali, saponi 30: 473–74. 1948.

85. Bohmi, H. Acidimetric method for determination of mustard oil. Pharm. Zentralhalle 87: 299–301. 1948.

86. Naves, Y.-R. Volatile plant materials. 61. Presence of nerolidol in essential oils of the *Papilionaceae*. Helv. Chim. Acta 31: 408–17. 1948.

87. Guillot, M. and Guillot-Allegre, S. Preservation of essential oils. Ann. pharm. franc. 6: 433–37. 1948.

88. Mitra, C., Rao, P. N., Bhattacharji, S. and Siddiqui, S. Chemical examination of nim blossoms (*Melia azadirachta flora*). J. Sci. Ind. Research (India) 6(2)B: 19–24. 1947.

89. Bruns-Rungi, G. *Ocimum canum* Sims, camphor basil. Pharmazie 3: 315–21. 1948. Ibid. 262–72.

90. Youngken, H. W. and Hassan, E. W., Jr. Studies on the camphor basil. *Ocimum Kilimandscharicum* Gürke. J. Am. Pharm. Assoc., Sci. Ed. 37: 360–63. 1948.

91. Kalinkevich, M. D. Effect of potassium on accumulation of essential oils in the leaves of *Ocimum canum*. Doklady Akad. Nauk. S.S.S.R. 60: 1363–65. 1948.

92. Dulou, R. and Pitard, P. Chemical and spectrographic study of the oil of *Ocimum gratissimum* L. from the Society Islands. Ann. pharm. franc. 5: 534–39. 1948.

93. Hickey, M. J. Investigation of the chemical constituents of Brazilian sassafras oil. J. Organ. Chem. 13: 443–46. 1948.

94. Shukis, A. J. and Wachs, H. Determination of safrole in the oil of *Ocotea cymbarum*. Anal. Chem. 20: 248–49. 1948.

95. Lahey, G. and Sutherland, M. D. The odor of *Opercularia aspera* (Goertn). Queensland Univ. Dept. Chem. Papers Vol. 1 no. 32. May 31, 1948. 3 pp.

96. Naves, Y.-R. The aldehyde fraction of essential oil of sweet orange. Perfumery Essent. Oil Record 38: 295–98. 1947.

97. Nicholls, J. J. Essential oil of orange, Palestinian. Perfumery Essent. Oil Record 38: 240–42. 1947.

98. Islip, H. T. and Major, F. Orange oils from Palestine. Bull. Imp. Inst. 45(1): 15–17, 1947; 46(2–4): 213–15, 1948.

99. Costa, A. F. and Cardoso do Vale, J. Analysis of the essential oil of *Origanum virens* Hollms. and Link. Relationship to the chemical composition of related species. Notícias Farm. (Port.) 1948. 11 pp.

100. Briggs, L. H. and Sutherland, M. D. A terpene-type essential oil from a fern (*Paesia scaberula*). Nature (London) 160: 333. 1947.

101. Narian, K. and Das Gupta, P. N. Chemical examination of Indian palmarosa oil. Indian soap J. 13: 259–63. 1948.

102. Subba Rao, T. V. *et al.* Palmarosa oil. I. Indian Soap J. 14: 159–64. 1948.

103. Bradley, C. E. The essential oil of *Pectis papposa*. Proc. Confer. Cultivation Drug Assoc. Econ. Plants in Cal. 1947. 263–67.

104. Gokhale, V. G., and Phalnikar, N. L. and Bhide, B. V. Chemical investigation of the fruits of *Piper longum*. J. Univ. Bombay 16A: 47–52. 1948.

105. United States. Bureau of Agricultural Economics. Garlic and mint for oil. TC-48 no. 1229. Dec. 17, 1948. Mint for oil. TC-49 no. 803. August 10, 1949.

106. United States. Bureau of the Census. FT 410. United States exports of domestic and foreign merchandise. Commodity by country of destination. Part 1. Calendar years 1947 and 1948. Washington, D. C.

107. Tornow, P. A. and Fischer, L. A study of the physical and chemical properties of natural Washington and Oregon peppermint oils. J. Am. Pharm. Assoc., Sci. Ed. 37: 76–78. 1948.

108. Bullis, D. E., Price, F. E. and Kirk, D. E. Relationship of maturing and weathering to yield and quality of peppermint oil. Oregon Agr. Expt. Station Bull. no. 458. Aug. 1948. 15 pp.

109. Halasz, A. and Katz, J. Production of essential oils in Palestine. Ind. parfum. 3: 212–14. 1948.

110. Schmidt, H. Peppermint oil. Occurrence of jasmone in essential oil of *Mentha piperita* L. Chem. Ber. 80: 538–46. 1947.

111. Bedoukian, P. Z. Occurrence of menthofuran in oil of peppermint (*Mentha piperita vulgaris* S.). J. Am. Chem. Soc. 70: 621–22. 1948.

112. Garnero, J., Benizet, L. and Igolen, G. Chemical composition of the essential oil of Brazilian peppermint (*Mentha arvensis* D. C. var. *piperascens* Malinvaud). Ind. parfum. 3: 353–59. 1918.

113. Cabrera, C. F. The essential oil of *Mentha piperita*. New color reactions. Anales fis. y quim. (Madrid) 13: 789–91. 1947.

114. Birkeli, H. Ecological studies on *Mentha piperita*. 1. Effect of mineral nutrients and of water. Medd. Norsk Farm. Selskap 10: 149–62, 163–71, 178–91, 199–208. 1948.

115. Briggs, L. H. and Sutherland, M. D. The essential oil of *Phyllocladus trichomanoides*. J. Org. Chem. 13: 1–9. 1948.

116. Dupont, G., Dulou, R. and Naffa, P. Sesquiterpene components of turpentine from maritime pine: caryophyllene and longifolene. Bull. soc. chim. France, mem. 15: 990–94. 1948.

117. Sorensen, N. A. and Bruun, T. The occurrence of a diterpene-camphor in Scots fir (*Pinus silvestris*). Acta Chem. Scand. 1: 112–13. 1947.

118. Haagen-Smit, A. J., Redemann, C. T. and Mirov, N. T. Composition of gum turpentine of Torrey pine. J. Am. Chem. Soc. 69: 2014–17. 1947.

119. Khrimian, A. Yield of essential oil from the parts of the plant *Praugos fernlacea* (L) Lindl. Akad. Nauk Armianskoi S.S.S.R. Bot. Sad. B 1948(6): 51.

120. Schmid, H. and Karrer, P. Constitution of radish. 1. Sulforaphen, a mustard oil of radish seed (*Raphanus sativus* L. var. *alba*). Helv. Chim. Acta 31: 1017–28. 1948. 2. Optically active 1-methylsulfoxide-buten-3-yl-cyanide as cleavage product of the glucosides in the seed of *Raphanus sativus* var. *alba*. Ibid. 1087–92.

121. Maranca, G. Perfume of the leaves of May roses. Ind. parfum. 3: 360–61. 1948.

122. Ratti, A. and Tuveri, F. Essential oils of the plants of Sardinia. Rosemary oil. Rend. seminar. facoltá sci. Univ. Cagliari 16(3–4): 201–8. 1946. (Pub. 1948).

123. Fernandez, O. and Abiniacar, J. Essential oils of Spanish sage. Farm. nueva (Madrid) 12: 161–68, 215–22. 1947.

124. Youngken, H. W. and Heaps, E. W., Jr. Studies on the leaf of *Salvia leucophylla*. J. Am. Pharm. Assoc., Sci. Ed. 37: 284–87. 1948.

125. India. Department of Commercial Intelligence and Statistics. Accounts relating to the sea-bourne trade and navigation of India. March 1948 and March 1949.

126. Matei, I. and Cocla, E. Oil from the needles of the conifers of Rumania. Bull. école polytech. Jassy 3(1): 416–25. 1948.

127. Penfold, A. R., Morrison, F. R. and McKern, H. Studies in the *Myrtaceae* and their essential oils. 1. The seasonal variations in yield and cineol content of *Melaleuca alternifolia* Cheel. Researches on Essent. Oils Australian Flora 1(1): 5–7. 1948.

128. Penfold, A. R., Morrison, F. R. and McKern, H. Studies in the physiological forms of the *Myrtaceae*. 2. The occurrence of physiological forms in *Melaleuca alternifolia* Cheel. Researches on Essent. Oils Australian Flora 1(1): 18–19. 1948.

129. Penfold, A. R., Morrison, F. R. and McKern, H. *Melaleuca viridiflora*: a potential source of nerolidol and linalool. Perfumery Essent. Oil Record 39: 141–42. 1948.

130. Hegnauer, R. Chemistry and morphology of Swiss *Thymus* forms. Ber. schweiz. botan. Ges. 58: 391–461. 1948.

131. Rao, P. S. A note on the aromatic principle of *Tylophora indica*. Proc. Indian Acad. Sci. 28A: 173–74. 1948.

132. Wallis, T. E. and Sanyal, P. K. Valerian, European and Indian: volatile oil content and alcohol-soluble extractive. Quart. J. Pharm. Pharmacol. 20: 272–81. 1947.

133. Falconnier, L. Seasonal variations of *Valeriana officinalis* root. Ber. schweiz. botan. Ges. 57: 122–31. 1917. (Pharm. Zentralhalle 87: 311. 1918.)

134. Basu, N. K. and Singh, G. B. Investigations on Indian medicinal plants. 2. *Vitex negundo*. Quart. J. Pharm. Pharmacol. 20: 136–37. 1917.

135. Neugebauer, H. The action of the distillate of witch hazel (*Hamamelis virginiana*) and of *Corylus avellana*. Pharmazie 3: 313–14. 1948.

136. Halpern, A. Ascaridol in oil of chenopodium. J. Am. Pharm. Assoc., Sci. Ed. 37: 161–65. 1948.

137. Halpern, A. An investigation of the assay of ascaridol. 2. The iodination of terpenes. J. Am. Pharm. Assoc., Sci. Ed. 37: 465–67. 1948.

138. Cardoso do Vale, J. Colorimetry of a reaction with ferrous sulfate adapted to determination of ascaridol. Notícias farm. (Port.) 1948.

139. Cardoso do Vale, J. Reaction of Cocking and Hymas applied in the colorimetric determination of ascaridol. Notícias farm. (Port.) 1948.

140. Fonseca, Ester de Camargo. Essential oil content of wormwood, *Artemisia absinthium*, grown in Brazil. Anais faculdade farm. e. odontol. univ. São Paulo 5: 65–70. 1947.

141. Lederer, E., Mercier, D. and Perot, G. The constituents of ambergris. 3. Structure of ambrein. Bull. soc. chim. France, mem. 1947, 345–49.

142. Jeger, O., Durst, O. and Ruzicka, L. Triterpenes. 117. The constitution of ambrein. Helv. Chim. Acta 30: 1859–60. 1947.

143. Mukherji, Debi. Synthesis of *cis*-apofenchocamphoric acid. Science and Culture 13: 388–89. 1948.

144. Huckel, W. and Kirschner, H. G. Changes of molecular structure in chemical reactions. 7. Ring expansion in the oxidation of camphene. Chem. Ber. 80: 41–47. 1947.

145. Angla, B. Thiourea-camphor complexes. Compt. rend. 224: 402–4. 1166–67. 1947.

146. Ghiorghiu, C. V. and Budeanu, C. Condensation of isothiocyanates with oximes. 6. The conversion of camphor oxime to the nitrile of α-campholenic acid. Rev. Stiintifica "V. Adamachi" 34: 182–84. 1948.

147. Sorm, F. and Urbanek, L. Terpenes. 4. Constitution of carotol (from oil of *Daucus carota*). Collection trav. chim. tchécoslov. 13: 49–56. 1948.

148. Dupont, G., Dulou, R. and Bulteau, G. Brominated derivatives of inactive carvomenthene. Bull. soc. chim. France, mem. 1948, 195–97.

149. Bordenca, Carl. Cinnamon oil substitutes. Am. Perfumer Essent. Oil Rev. 49: 47–48. 1947.

150. Stoll, M., Hulstkamp, J. and Rouvé, A. Syntheses of macrocyclic products having a musk odor. 8. Synthesis of natural civetone. Helv. Chim. Acta 31: 543–53. 1948.

151. Briggs, L. H. and Taylor, W. I. Constitution of copaene. J. Chem. Soc. 1947, 1338–39.

152. Gillespie, D. T. C., Macbeth, A. K. and Mills, J. A. Reactions of α, β-unsaturated cyclic aldehydes and ketones. 6. (+)-*Cis*- and (−)-*trans*-cryptol from (−)-cryptone. J. Chem. Soc. 1948, 996–99.

153. Pigulevskii, G. V. and Markina, G. V. Asymmetric synthesis in linalool derivatives. Doklady Akad. Nauk. S.S.S.R. 63: 677–80. 1948.

154. Meijer, T. M. and Schmid, H. Constitution of eugenine. Helv. Chim. Acta 31: 1603–7. 1948.

155. Schmid, H. and Meijer, T. M. The constitution of eugenone. Helv. Chim. Acta 31: 718–52. 1948.

156. Wright, S. E. Constitution of evodione. J. Chem. Soc. 1948, 2005–8.

157. Huckel, W. Changes of molecular structure in chemical reactions. 6. Transformation of α-fenchylamine with nitrous acid. Chem. Ber. 80: 39–41. 1947.

158. Huckel, W. and Kindler, H. Changes of molecular structure in chemical reactions. 8. A new fenchene, ξ-fenchene. Chem. Ber. 80: 197–202. 1947.

159. Alquier, René. Some observations noted in the course of the dehydrogenation of geraniol on a reduced copper catalyst. Ind. parfum. 2: 405–6. 1947.

160. Challis, A. A. L. and Clemo, G. R. Homovanillin. J. Chem. Soc. 1947, 1692–97.

161. Buu-Hoi, NG Ph. and Royer, R. Influence of steric hindrance in indole syntheses according to E. Fischer. Rec. trav. chim. 66: 305–11. 1947.

162. Naves, Y.-R. Volatile plant materials. 46. Resolution of *d,l*-α-ionone. Helv. Chim. Acta 30: 769–74. 1947.

163. Henbest, H. B. Synthesis of dehydro-β-ionone. Nature 161: 481. 1948.

164. Karrer, P. and Ochsner, P. Dehydrogenation of β-(and α-) ionone by means of bromosuccinimide. Helv. Chim. Acta 31: 2093–97. 1948.

165. Ruzicka, L., Buchi, G. and Jeger, O. Violet perfumes. 24. Synthesis of dihydro-γ-ionone, a degradation product of ambrein. Helv. Chim. Acta 31: 293–95. 1948.

166. Ruzicka, L., Seidel, C. F. and Pfeiffer, M. The volatile constituents of ambergris. 1. Isolation of dihydro-γ-ionone. Helv. Chim. Acta 31: 827–31. 1948.

167. Koster, H. Perfumes of the ionone group. 5. Condensation of citral with ketones. Chem. Ber. 80: 248–55. 1947.

168. Prelog, V. and Frick, H. Violet perfumes. 25. Two diastereoisomeric tetrahydro-ionanes. Helv. Chim. Acta 31: 417–22. 1948.

169. Prelog, V., Fuhrer, J., Hagenbach, R. and Schneider, R. Organ extracts. 13. Isolation of ionone derivatives from the urine of pregnant mares. Helv. Chim. Acta 31: 1799–1811. 1948.

170. Prelog, V. and Frick, H. Violet perfumes. 28. Ketotetrahydro-ionone. Helv. Chim. Acta 31: 2135–42. 1948.

171. Lederer, E., Prelog, V. and Schneider, R. Organ extracts. II. The presence of ionone derivatives in castoreum. Helv. Chim. Acta 31: 2133–35. 1948.

172. Ruzicka, L., Seidel, C. F. and Brugger, W. Violet perfumes. 17. The degradation of tetrahydro-irone. Helv. Chim. Acta 30: 2168–98. 1947.

22. The degradation of dihydro-irone and dihydro-irane. L. Ruzicka and C. F. Seidel. Ibid. 31: 160–72. 1948.

173. Schappi, G. and Seidel, C. F. Violet perfumes. 18. Some syntheses in the field of degradation products of tetrahydro-irone. Helv. Chim. Acta 30: 2199–2212. 1947.

174. Naves, Y.-R. Volatile plant materials. 53. Contribution to the knowledge of irone. Helv. Chim. Acta 30: 2222–32. 1947.

54. Spectral absorption and the Raman spectrum of irone, dihydro-irol and related substances. Y.-R. Naves and P. Bachmann. Ibid. 2233–40.

175. Ruzicka, L. *et al.* Violet perfumes. 15. The constitution of irone. Helv. Chim. Acta 30: 1807–10. 1947.

23. Supplementary investigations on the constitution and synthesis of irone. Ibid. 31: 257–81. 1948.

176. Naves, Y.-R. Volatile plant materials. 62. Constitution and synthesis of irone. Helv. Chim. Acta 31: 893–914. 1948.

177. Naves, Y.-R. Volatile plant materials. 73. The *cis* (2, 6)-*d*-γ-irone of which the 4-phenylsemicarbazone melts at 178–9°. Helv. Chim. Acta 31: 2047–51. 1948.

178. Schinz, H., Ruzicka, L., Seidel, C. F. and Tavel, C. Violet perfumes. 16. Synthesis of *d,l*-α-irone. Helv. Chim. Acta 30: 1810–11. 1947.

179. Naves, Y.-R., Grampoloff, A. V. and Bachmann, P. Volatile plant materials. 50. The 3-methyl linalools. 3-methyl citrals and 6-methyl ionones. Helv. Chim. Acta 30: 1599-1613. 1947.

180. Naves, Y.-R. Volatile plant materials. 64. Synthesis of *cis*-(2,6)-*d,l*-α-irone. Helv. Chim. Acta 31: 1103-11. 1948.

67. Separation and isolation of the α- and γ-irones and a hitherto unknown α-irone. Ibid. 1280-88. 1948.

181. Naves, Y.-R. Volatile plant materials. 70. The presence of neo-α-irone in iris. Helv. Chim. Acta 31: 1876-78. 1948.

182. Naves, Y.-R. Volatile plant materials. 69. Tetrahydro-irones. Helv. Chim. Acta 31: 1871-76. 1948.

183. Winter, M., Schinz, H. and Stoll, M. Violet perfumes. 19. Synthesis of 4-methyl ionone. Helv. Chim. Acta 30: 2213-15. 1947.

184. Rouvé, A. and Stoll, M. Violet perfumes. 20. Synthesis of 5-methyl ionone. Helv. Chim. Acta 30: 2216-20. 1947.

185. Naves, Y.-R. and Ardizio, P. Voltile plant materials. 75. Synthesis of tetramethylcyclohexenyl formaldehydes in connection with the preparation of the iso-irones. Helv. Chim. Acta 31: 2252-56. 1948.

76. Synthesis of irone isomers. Ibid. 2256-62.

186. Briggs, L. H. and Cawley, R. W. Diterpenes. 1. Identity of kaurene with podocarprene. J. Chem. Soc. 1948, 1888-89.

187. Ghiorghiu, C. V. and Budeanu, C. The condensation of isothiocyanates with oximes. 7. The action of benzoyl isothiocyanate on cyclic ketoximes. Ann. sci. univ. Jassy 31, Pt. 1. 16 pp. 1948.

188. Naves, Y.-R. and Korpi, U. Volatile plant materials. 49. Polymorphism of *d,l*-menthyl oxalate. Helv. Chim. Acta 30: 1219-23. 1947.

189. Prelog, V. and Meier, H. L. The two optically inactive forms of *d,l*-menthyl oxalate described by Y.-R. Naves and U. Korpi. Helv. Chim. Acta 31: 177-79. 1948.

190. Stoll, M. and Hulstkamp, J. Synthesis of macrocyclic products having a musk odor. 2. An improvement in the preparation of cyclic acyloins. Helv. Chim. Acta 30: 1815-21. 1947.

3. Cyclic acyloins. M. Stoll and A. Rouvé. Ibid. 1822-36.

4. The transformation of cyclanolones to cyclanones. M. Stoll. Ibid. 1837-45.

9. Transformation of 2-cyclopentadecen-1-one to muskone. M. Stoll and A. Commarmont. Ibid. 31: 554-55. 1948.

10. Transformation of 1,2-cyclohexadecanediol to 1,2-epoxycyclohexadecane and cyclohexadecanone. Ibid. 1077-81.

11. Reduction of 15-methyl-2,1-cyclopentadecanolone to α-methylcyclopentadecanone. M. Stoll. Ibid. 1082-86.

12. Reduction of β-methylcyclopentadecanoin to muskone and γ-methylcyclopentadecanone. M. Stoll and A. Commarmont. Ibid. 1435-38.

191. Prelog, V. *et al.* Carbon rings. 43. Method of preparing many-membered cyclanones. Helv. Chim. Acta 30: 1741-49. 1947.

192. Crabalona, L. Bromine number and maleic complex of ocimene. Bull. soc. chim. France, mem. 1948, 384-88.

193. Crabalona, L. Presence of β-ocimene in flower and leaf oils of the bitter orange of France. Bull. soc. chim. France, mem. 1948, 388-90.

194. Haagen-Smit, A. J. and Fong, C. T. O. Chemical investigations in guayule. 2. The structure of partheniol, a sesquiterpene alcohol from guayule. J. Am. Chem. Soc. 70: 2075-79. 1948.

195. Berry, P. A. *l*-β-Phellandrene. Australian Chem. Inst. J. & Proc. 11: 387–90. 1947.

196. Berry, P. A. and Macbeth, A. K. β-Phellandrene tetrabromide. J. Chem. Soc. 1947. 1039–41.

197. Acheson, G. G. and West, T. F. Reaction of β-naphthol with *l*-α- and *l*-β-phellandrene. Nature 161: 976. 1948.

198. Chaudhuri, Piyus Kanti. A new synthesis of *d,l*-piperitone. Science and Culture 13: 348. 1948.

199. Karrer, P. and Ochsner, P. 2,2,6-Trimethyl-3,5-cyclohexadienal. Helv. Chim. Acta 30: 2092–95. 1947

200. Briggs, L. H. and Taylor, W. I. Cadalene and eudalene trinitrotoluates. J. Am. Chem. Soc. 69: 716. 1947.

201. Gurgen, Ali. Investigations on important Turkish essential oils. Ankara Yüksek Zir. Enstitüsü Derg. 9: 332–60. 1948.

202. Chopra, I. C., Handa, K. L. and Kapoor, L. D. A preliminary note on the essential oil bearing plants growing in Kashmir. 2–3. Indian J. Agr. Sci. 17: 100–3, 389–92. 1947.

203. Langlais, P. and Bollinger, J.-L. Research in the field of natural perfumes. Ind. parfum. 2: 13–19, 78–82, 123–28. 1947.

204. Brasch, Arno (to Electronized Chemicals Corp.). Method of making scenting essential oils from plants. U. S. pat. 2,457,203. 1948.

205. Weichan, Cilly. The essential oil content of aromatic plants in relation to fertilizer. Pharmazie 3: 464–67. 1948.

206. McKern, H. H. G. and Smith-White, S. An improved distillation apparatus for the determination of the essential oil content of plant material. Australian Chem. Inst. J. & Proc. 15: 276–78. 1948.

207. Burdick, Everett M. and Allen, James S. Rapid estimation of citrus peel oil. Anal. Chem. 20: 539–41. 1948.

208. Caujolle, F. and Couturier, P. Solubility of essential oils in aqueous solutions of sodium thymotate. Ind. parfum. 3: 240–41. 1948.

209. Hayward, F. W. and Seymour, R. B. Determination of major constituents of cedar oil vapor in cedar chests. Anal. Chem. 20: 572–74. 1948.

210. Navis, Y.-R. Volatile plant materials. 17 and 51. Estimation of alcohols in essential oils by acetylation in pyridine. Helv. Chim. Acta 30: 796–800, 1613–15. 1947.

211. Gottlieb, O. R. New rapid method for the determination of linalool. Rev. quím. ind. (Rio de Janeiro) 16(188): 5–11. 1947.

212. Scholtens, C. Determination of carbonyl compounds in essential oils by means of 2,4-dinitrophenylhydrazine. Perfumery Essent. Oil Record 38: 235–37. 1947.

213. Lombard, R. Raman spectra of some terpene oxides. Bull. soc. chim. France, mem. 1947. 522–26.

214. Navis, Y.-R. and Ardizio, P. Volatile plant materials. 66. Ultraviolet absorption of terpene derivatives and aliphatic sesquiterpenes. Helv. Chim. Acta 31: 1210–16. 1948.

215. Navis, Y. R. Volatile plant materials. 71. Ultraviolet absorption spectra of solutions of irone in iso-octane. Helv. Chim. Acta 31: 2057–60. 1948.

216. Gunthard, H. and Ruzicka, L. Violet perfumes. 27. Infrared spectra in the irone and ionone series. Helv. Chim. Acta 31: 642–52. 1948.

217. Svem-Moldavskii, G. Ya. Action of vapors of ethereal oils on one-celled organisms. Byull. Eksp. Biol. i Med. 23: 292–96. 1947.

218. Piacentini, E. Antiseptic and disinfectant power against sporogens of bergamot, orange and lemon oils in aqueous solution. Ann. igiene 58(1): 1–12. 1949.

219. Klosa, J. Some plant substances that inhibit germination of seeds and growth of bacteria. Pharmazie 3: 410–13. 1948.

220. Guillot, M. Electrical behavior of vapors of odoriferous organic materials in air. Compt. rend. 224: 39–41. 1947.

221. Guillot, M. Partial anosmia and fundamental odors. Compt. rend. 226(16): 1307–9. 1948.

222. Guillot, M. Relation between odor and molecular structure. Compt. rend. 226: 1172–74. 1948.

223. Le Magnen, J. Analysis of complex homologous odors by the fatigue method. Compt. rend. 226(9): 753–54. 1948.

224. Le Magnen, J. A case of olfactory sensitivity presented as a female secondary sex characteristic. Compt. rend. 226: 694–95. 1948.

225. Le Magnen, J. Olfactive excitation. Compt. rend. 225: 1378–80. 1947.

226. France. Direction Generale des Douanes. Statistique mensuelle du commerce extérieur de la France. Dec. 1947 and Dec. 1948.

227. Spain. Direccion Gen. de Aduanos. Estadística del Comercio exterio de Espana. 1947 and 1948.

228. Switzerland. Zollverwaltung. Monatsstatistik des Aussenhandels der Schweiz. Jan.–Dec. 1947; Jan.–Dec. 1948.

229. United Kingdom. Accounts relating to trade and navigation of the United Kingdom. Dec. 1947; Jan.–Dec. 1948.

230. United States. Bureau of the Census. FT 110. United States imports for consumption of merchandise. Commodity by country of origin. Calendar years 1947 and 1948. Washington, D. C.

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